

Xenon Hyperpolarizer

Model 9800

**Operating and Service
Manual**

Version 1.2

Part # 03662

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Xenon Polarizer Operating and Service Manual

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TABLE OF CONTENTS

1.	Introduction	
	Using This Manual	1
	Chapter Guide	2
	Document Conventions	3
	Abbreviations	3
	Revision History	8
2.	Safety and Precautions	
	EMERGENCY SHUTDOWN INSTRUCTIONS	1
	Warnings and Precautions	1
	Some Common Wisdom	2
	Revision History	4
3.	Operational Requirements	
	Operator Requirements	1
	Equipment Requirements	1
	Utility Requirements	1
	Revision History	2
4.	Polarizer Specifications	
	Polarizer Description	1
	Cartridge Components	1
	Performance Specifications	2
	Polarization Cell	3
	Cold Finger	4
	Typical Settings	5
	Revision History	6
5.	Installation	
	Plumbing and Electrical Connections	1
	Cold Finger Installation	4
	Optical Cell Installation	4
	Finishing the Installation	5
	Revision History	6
6.	Operating the Polarizer	
	Powering Up the Polarizer	1
	Turning the Laser On and Off	2
	Turning On the Laser	2
	Turning Off the Laser	3

Leaving the Polarizer in Stand-By Mode	3
Shutting Down the Polarizer	3
Revision History	4
7. Polarizing Xenon	
Initializing the Xenon Collection	1
Collecting Hyperpolarized Xenon	4
Terminating the Collection	5
Thawing Frozen Xenon	6
Preparing a Consecutive Batch	8
Revision History	10
8. Cold Finger Operations	
Detaching the Cold Finger	1
Attaching the Cold Finger	1
Revision History	2
9. The Temperature-Interlock Unit	
Controlling Cell Temperature	2
Connecting an External Interlock (Optional)	2
Revision History	2
10. Cleaning and Transporting the Polarizer	
Cleaning the Polarizer	1
Moving the Polarizer	1
Preparing the Polarizer for Moving	2
Shutting Down the Polarizer	2
Revision History	3
11. Maintenance Procedures	
Maintenance Schedule	2
Plumbing Layout	2
Protective Housing Maintenance	4
Checking for the Presence and Integrity of Protective Housing	4
Verifying Beam Path Protective Housing Integrity	4
Replacement of Protective Housing Gaskets	5
Vacuum Pump Maintenance	6
Checking the Oil Level/Condition in the Vacuum Pump	6
Changing the Oil in the Vacuum Pump	6
Optical Cell Maintenance	7
Depressurizing the Optical Cell	7
Pressurizing the Optical Cell	8
Checking the Condition of the Optical Cell	8
Replacing the Optical Cell	9
Gas Manifold Maintenance	11

Replacing the Xenon Mixture Tank	11
Replacing the UHP Nitrogen Tank	12
Regenerating the Gas Purifier	13
Cleaning the Cold Finger	14
Rinsing the Cold Finger	14
Revision History	16

APPENDICES

12. A. Using the Computer Applications

The Laser Control Application (IntegraSoft) for Systems with Spectra-Physics Lasers	1
Starting the Interface	2
Starting Laser Emission	3
The Polarimetry Application (version 4.1 and above)	5
Navigating the Data Acquisition View	5
Gas Sample Settings: Pressure, Blend, and Pulse Parameters	6
Performing Data Acquisitions	7
Notifying the Operator of Uncertainty in Calculated Values	7
Increasing Signal-to-Noise for Low Amplitude Signals	7
Recording the Baseline Transmission	9
Viewing Polarimetry Inventory Information	9
Viewing Polarimetry Configuration	10
Polarimetry Error Messages	11
Revision History	12

13. B. Troubleshooting

Revision History	4
------------------------	---

14. C. Accumulation Data Sheet

Revision History	1
------------------------	---

15. D. Polarizer Interlocks

Removing and Replacing Interlocked Panels	2
Cart End Panels	2
Oven Covers	2
Interlock Troubleshooting	3
Revision History	4

16. E. Rubidium MSDS

1

Introduction

Welcome to Polarean's *9800 Xenon Polarizer*. Using the Xenon polarizer, a trained operator can polarize ^{129}Xe by completing the following steps, described briefly below:

1. The operator, through choice of gas flow rate and accumulation time, determines gas quantity and production rate.
2. During production, an input mixture containing ^{129}Xe continuously flows from the storage tank to the polarization chamber, where it is polarized via spin exchange optical pumping.
3. Upon exiting the chamber, polarized Xenon is frozen into the liquid nitrogen-cooled cryogenic accumulator while residual He and N_2 in the mixture are vented into the atmosphere.
4. After the desired quantity of solid Xenon has been accumulated, the Xenon is sublimated and vented into an appropriate container.

These steps are explained in detail in the rest of this manual.

Note: A trained operator must start, stop, and periodically monitor the polarization process.

Using This Manual

The *Xenon Polarizer Operating and Service Manual* provides step-by-step instructions for installing and servicing the Xenon polarizer, as well as performing all standard user procedures. These tasks include:

- Polarizer specifications
- Operational requirements
- Installation procedures

- Powering up the polarizer
- Turning the laser on and off
- Polarizing, collecting, and thawing Xenon
- Basic maintenance procedures

Chapter Guide

This manual is divided into the following chapters:

- **Chapter 1**, Introduction — introduces the Xenon Polarizer and explains how to use this manual
 - **Chapter 2**, Safety and Precautions — explains emergency shutdown procedures for the polarizer
 - **Chapter 3**, Operational Requirements — lists all operational requirements for the polarizer
 - **Chapter 4**, Polarizer Specifications — gives a summary of the polarization process and describes each component
 - **Chapter 5**, Installation — lists step-by-step procedures for installing the polarizer, the cold finger, and the optic cell
 - **Chapter 6**, Operating the Polarizer — lists instructions for powering up the polarizer and turning the laser on and off
 - **Chapter 7**, Polarizing Xenon — walks through the polarization process from start to finish
 - **Chapter 8**, Cold Finger Operations — documents how to attach and detach the cold finger
 - **Chapter 9**, The Temperature-Interlock Unit — explains how to control the cell temperature
 - **Chapter 10**, Cleaning and Transporting — provides instructions for how to move the polarizer, if necessary
 - **Chapter 11**, Maintenance Procedures — explains the periodic procedures needed to maintain the polarizer
 - **Appendix A**, Using the Computer Applications — describes how to use the software applications that accompany the polarizer
 - **Appendix B**, Troubleshooting — provides solutions to common problems
 - **Appendix C**, Accumulation Data Sheets — for recording accumulation statistics
-

- **Appendix D**, Polarizer Interlocks — explains how the polarizer interlocks function
- **Appendix E**, Rubidium MSDS— describes general properties of Rb alkali metal

Document Conventions

Emphasized text appears in italics. For example:

- *Hand tighten* the QF16 flange clamp over the connection.
- *Do not operate the polarizer until all sources of excessive emissions have been repaired.*

A note or an example contains information that is helpful, but not crucial to the task at hand. For example:

Note: The indicator LED should be green.

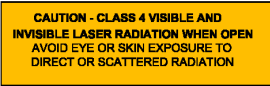
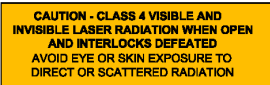
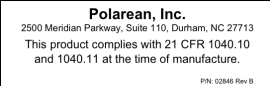
Example: Typing *S* in the name box does not yield all names beginning with S.

A warning contains information that, if ignored, may harm the operator or the equipment. A list of standard warning symbols used in this manual is provided below.

Warning Symbols

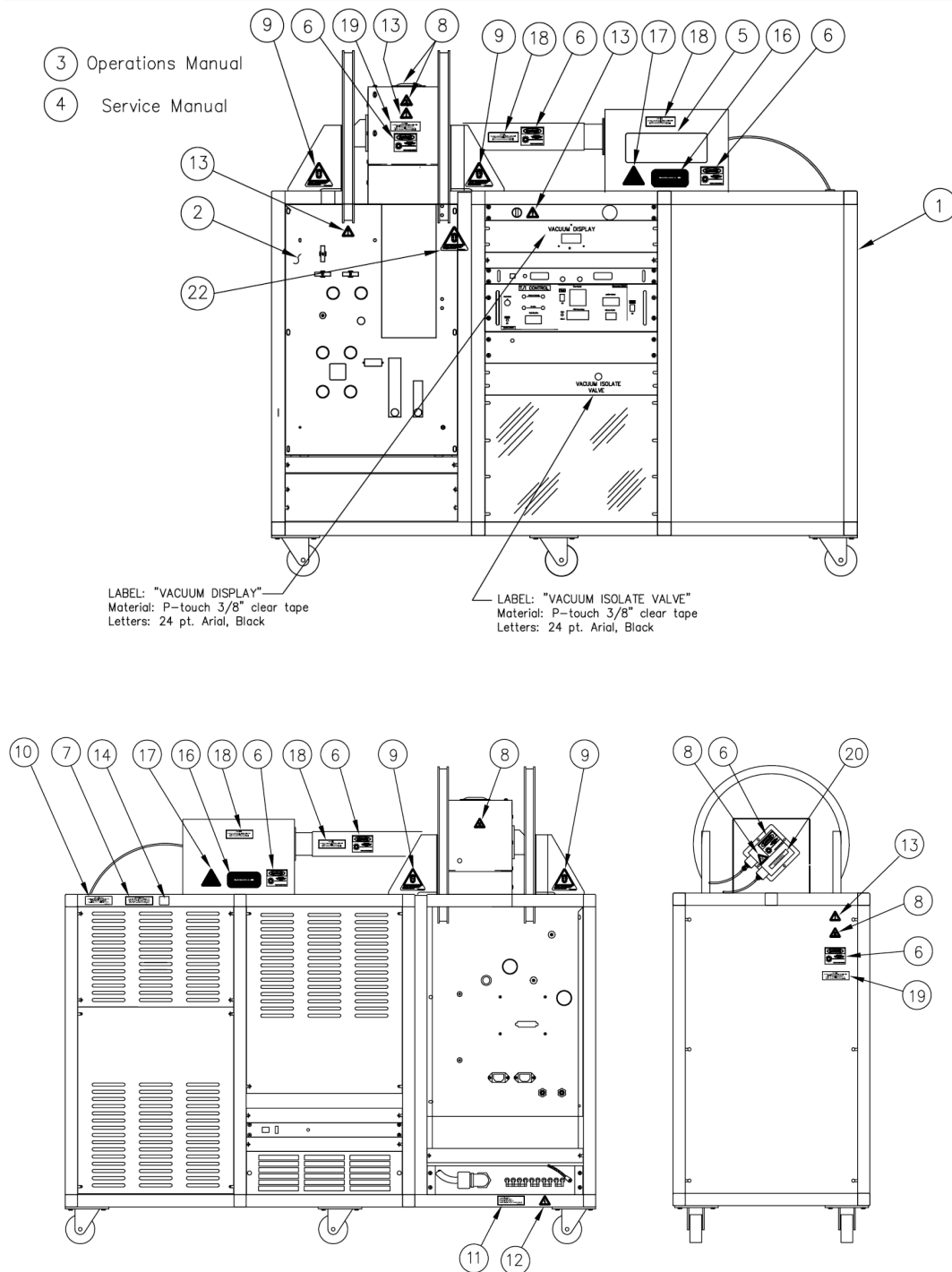
The following warning symbols may be used in this manual and/or on the unit:

-  This symbol is intended to alert the operator to the presence of important operating and maintenance instructions.
-  This symbol is intended to alert the operator to the presence of hot surfaces that may cause burns to the skin.
-  This symbol is intended to alert the operator to the presence of dangerous voltages that may be of sufficient magnitude to constitute a risk of electric shock.
-  This symbol is intended to alert the operator to the danger of exposure to hazardous visible and invisible laser radiation.

- 5  This symbol is intended to alert the operator to the danger of exposure to magnetic fields.
- 6  This symbol is intended to alert the operator to the presence of hazardous laser radiation inside the unit.
- 7  This is the laser product explanatory label and IEC 60825-1 conformance declaration.
- 8  This symbol is intended to alert the operator to the danger of exposure to hazardous visible and invisible laser radiation if the housing is open during use.
- 9  This symbol is intended to alert the operator to the danger of exposure to hazardous visible and invisible laser radiation if the housing is open and the protective interlock is defeated.
- 10  This symbol is intended to alert the operator to the danger of exposure to hazardous visible and invisible laser radiation if the housing is open during use.
- 11  This is the declaration of conformance of the product to 21 CFR 1040.10 and 1040.11 at the time of manufacture.
- 12  This symbol is intended to alert the operator that the timing or precision of a specific step in the operating procedures is critical to achieving a high polarization yield or obtaining an accurate estimate of polarization level.

Warning Symbol Locations

The diagrams on the next page show the location of warning labels affixed to the Xenon polarizer.



Abbreviations

The following table lists the abbreviations and acronyms that may be used in this manual.

Table 1: Abbreviations and acronyms

Abbreviation/ Acronym	Meaning
C	Celsius
μ W	microwatt
atm	atmosphere
cc	cubic centimeter
cm	centimeter
dB	decibel
G	Gravitational acceleration
ID	Inner Diameter (or inside diameter)
kW	kilowatt
LED	Light-Emitting Diode
MSDS	Material Safety Data Sheet
mTorr	milliTorr
nm	nanometer
NMR	Nuclear Magnetic Resonance
OD	Outer Diameter (or outside diameter)
OFHC	Oxygen-Free High Conductivity
PFA	PerFluoroAlkoxy (a material similar to Teflon)
psi	Pounds per square inch
psig	Pounds per square inch gauge
PTFE	Poly Tetra Fluoro Ethylene (aka, DuPont Teflon)
Rb	Rubidium
RTD	Resistance Temperature Detector

Abbreviation/ Acronym	Meaning
SCFH	Cubic feet per hour at standard conditions
SCFM	Cubic feet per minute at standard conditions
slm	Standard liters per minute
torr	A unit of pressure equal to 1/760 of an atmosphere (about 133.3 pascals)
UHP	Ultra High Purity
UPS	Uninterruptible Power Supply
V	Volts
VAC	Volts of alternating current
VDC	Volts of direct current
Xe	Xenon
¹²⁹ Xe	The isotope of Xenon with atomic weight 129

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2

Safety and Precautions



All operators must read this guide *before* operating the polarizer or any piece of equipment contained within.

Operation of the Xenon polarizer without the proper training is prohibited and could cause irreversible damage to the individual, the facility, and the polarizer.

EMERGENCY SHUTDOWN INSTRUCTIONS

To shut down the polarizer, press the red Emergency Off button on the power distribution panel (at the top center of the polarizer).



If the optional uninterruptible power supply (UPS) is installed, the emergency shutdown will *not* cut power to the computer, monitor, or any other device connected to the on-board UPS. *As long as the UPS is on, 120 VAC is provided to these systems.*

To shutdown the UPS and all connected devices, press the Off button on the UPS front panel.

Warnings and Precautions



The Xenon Polarizer contains an enclosed Class 4 laser system. The removal of any portion of the Polarizer is *prohibited* during normal operation. Visible and/or invisible laser radiation exposure can occur when the protective housing is removed. Avoid eye and/or skin exposure to direct or scattered radiation.

- Keep all laser-shielding apparatus and housing in place when operating the system.
- Operators should never attempt to remove any portion of the polarizer affixed with tamper-resistant (safety) screws. If a fault is suspected in a component located behind any cover affixed with tamper-resistant screws, please contact Polarean.

- All operators of the polarizer must have been trained by Polarean in proper usage and safety precautions.
- Only a trained operator *must* perform any service or installation of the Xenon Polarizer.
- The Xenon Polarizer contains glass vessels under high pressures (routinely up to 75 psig but can be higher in case of misuse or gas regulator failure). Protective eyewear should be worn any time a panel is removed from the unit.
- The optical cell and cold finger are glass and cannot withstand rapid or extreme pressure changes. When pressurizing either the cell or cold finger, open the valves slowly. Failure to do so may result in a glassware integrity failure.



Never over-tighten the **Flow Control** valve as it can be damaged.

- If the optical cell explodes, close the Cell Isolate and Xe mixture tank valves immediately to prevent rapid loss of Xe mixture gas. Contact Polarean immediately.
- The cryogen access door must remain closed and latched any time the cold finger is pressurized (as evidenced by the digital readout below and to the left of the cryogen access door), except for when thawing the frozen Xenon.



The Xenon polarizer is equipped with a very strong permanent magnet installed on both sides of the cold finger. The cryogen access door must remain closed when the polarizer is not in use. Be extremely cautious when moving tools or other metal objects near the cold finger.

- Wear eye protection while thawing and dispensing gas and while the cryogen access door is open.



Some components are very hot and are marked as such. Do *not* touch the oven, laser beam enclosure housing, or any surface marked as Hot while the polarizer is on.

- Polarean recommends having a dry chemical (powder) fire suppressant in the same room as the polarizer (do not use a pressurized extinguisher as this could simply spread the burning rubidium metal). The rubidium metal in the optical cell will ignite explosively if exposed to water or foam.

Some Common Wisdom

- Do not close valves too tightly; use two fingers to close valves to prevent damage to the seals.
- When checking to see if a valve is open or closed, always try to *close* it. This will eliminate any accidental openings.

- Always wear eye protection when you are working with any part of the cell or cryogenic collection assemblies. (Eye protection is provided by Polarean.)
 - Never disable or remove any of the safety or laser intensity reduction mechanisms (laser safety windows, laser interlock, shielding apparatus). Permanent, severe injury may result.
 - Verify that all safety mechanisms and shielding components are in place and working before you turn the laser on.
 - Each internal component is connected to a central grounding bar via a grounding strap. Disconnect the grounding strap from the bar before removing any component.
-

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3

Operational Requirements

Operator Requirements



All operators must have read this guide *before* operating the polarizer or any of its components.

- The polarizer contains glassware under pressure. All glassware should be depressurized before moving or performing any maintenance on the polarizer.
- Operators must be trained in the proper handling of cryogenic liquids and have the appropriate safety equipment.



All on-site maintenance personnel must be trained by certified Polarean personnel in order to maintain the polarizer. Hazardous Class 4 laser exposure could otherwise result.



The key(s) that control the laser power at the control panel should be accessible only to trained operators and should be removed from the control panel when the laser is not in use.

Utility Requirements

The installation site should meet the following utility requirements:

- Minimum footprint required for the Xenon polarizer is $\sim 9.5' \times 7'$. A minimum of 2' of space is required between the polarizer and any wall and at least 3 feet of space in front of the polarizer.



Ferrous materials must be kept at least 3' from the polarizer.

- Room air conditioning required with a minimum cooling capacity of 18,000 BTUH. The air conditioning unit should be capable of maintaining the room temperature at $22\pm 2^{\circ}\text{C}$ (68–75°F) with a maximum relative humidity of 50%. The polarizer room should have its own dedicated temperature/humidity control system.
- Source of compressed air with a minimum required pressure of 20 psi capable of providing a minimum of 4 SCFM air flow, preferably through a $\frac{1}{4}$ " pipe fitting. The air supply moisture content should be less than 0.01%.
- Power outlet required at 3Ø208V US standard (47/63 Hz) rated at 20 A per phase with neutral, and equipped with a receptacle type NEMA L21-30R.

Installation Requirements

The following items are required for installation of the Xenon polarizer:

- Wall-mount cylinder holder rack with capacity to house three compressed gas cylinders
- Dry nitrogen gas cylinder
- UHP nitrogen or helium (99.995%) gas cylinder
- Xenon gas mixture cylinder (1% xenon recommended)
- Two UHP grade CGA 580 pressure regulators and one standard two-stage CGA 580 pressure regulator along with the necessary fittings and tubes to connect the three compressed gas cylinders to the polarizer. If a *Xenon Gas Manifold* is procured along with the polarizer, this item will not be required.

Operating Requirements

- Safety glasses
- Laser attenuating eyewear ($\text{OD} > 4$ at 795nm) for *each* maintenance technician
- 4-L liquid nitrogen transfer dewar
- Synthetic vacuum oil (1 gal)

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4

Polarizer Specifications

This chapter lists the various components of the polarizer, their descriptions, their physical makeup, and their performance specifications.

Polarizer Description

The following list describes the physical components of the polarizer:

- A ^{129}Xe polarization cartridge that fits securely in the left compartment of polarizer cart assembly.
- A polarizer cart assembly housing the ^{129}Xe polarization cartridge and other components as follows:
 - Line-narrowed laser and optical conditioning box
 - Temperature and interlock (T/I) controller
 - Polarimeter and light transmission monitor
 - Helmholtz coils and variable power supply
 - Vacuum pump
 - Computer
 - Power distribution

Cartridge Components

The following list describes the components that compose the ^{129}Xe polarizer cartridge:

- Plumbing within the cartridge that is leak tested to $\leq 5 \times 10^{-6}$ standard cc's of He per second, and is clean to "O₂ service" grade or better.
-

- A heat-activated gas purifier in the gas flow path that removes trace H₂O, O₂, CO, and CO₂. The purifier is able to accommodate a total gas flow rate of 5 L/min.
- Components for handling polarized ¹²⁹Xe composed of non-ferrous materials that are largely free of paramagnetic contaminants. Examples of materials used include: Pyrex, Aluminum (non-anodized), PTFE, PFA, silver-plated materials, gasket materials, and other high-purity non-paramagnetic coatings. Xenon outlet port connection accepts 1/4" OD tubing (user supplied).
- Mass flow meter with 5% accuracy and 0–4 L/min operating range.
- Pressure transducer with ±5% accuracy and –15 to 250 psig operating range.
- Shielding which protects the operator from cryogenics and high-pressure glassware during normal operation. An oven capable of sustained air temperatures of 200° C.
- At least 2 RTDs within the oven for measurement of air temperature and cell surface temperature.

Performance Specifications

Polarization is a function of gas flow rate (F_{rate}), laser power, laser light absorption, gas pressure, total accumulated Xe volume (V_{Xe}), and thawing technique.

The volume of the outlet manifold for handling polarized ¹²⁹Xe is 52 cm³. The outlet manifold volume includes the cold finger and is bounded by the following valves (all closed): *General Isolate* and *Flow-Shut-Off* with ¹²⁹Xe Outlet capped. The remaining valves (*Xe Out* and *Flow Isolate*) are open.

To calculate the collection time per volume, use the following formula:

$$V_{\text{Xe}} [\text{cc}] = F_{\text{rate}} [\text{sccm}] \cdot \text{Fraction}_{\text{Xe}} [\%] \cdot T_{\text{accumulation}} [\text{min}]$$

Example: 250 cc of ¹²⁹Xe = (1000 cc/min) · (0.01% Xe) · (25 min)

See **Figure 1** for a set of representative performance curves.

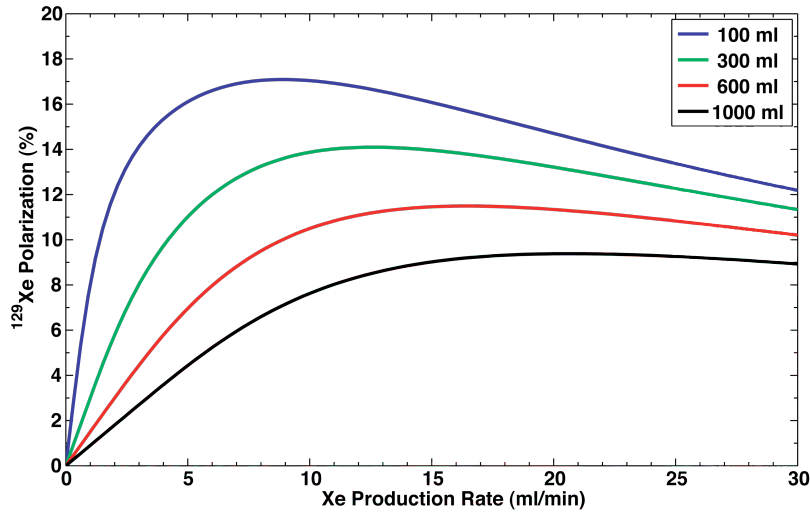


Figure 1: ^{129}Xe polarization vs. flow rate curve

Optical Polarization Cell

The polarization cell is shown in **Figure 2** and has the following properties:

- The cell is made of Pyrex, with O-rings made of Ethylene-propylene and Viton; maximum pressure rating: 8 atm
- Approximate optical cell length: 13 cm
- Approximate optical cell diameter: 5 cm
- Approximate optical cell volume: 250 cc
- Rb content >50 mg or sufficient to cause $\geq 50\%$ laser absorption for 6 atm gas mix pressure with a cell surface temperature at or below 200°C
- Rb purity $\geq 99.0\%$
- Inside the cell is an ultra-pure environment sealed off from exposure to O_2 , H_2O and other contaminants that could degrade Rb purity.
- Visible quantity of Rb located in the “Getter Bulb” in inlet arm of optical cell
- Maximum temperature to maintain 60% absorption: $T \leq 200^\circ\text{C}$.
Temperatures beyond this point will cause the Rb to ablate at an excessive rate and can deteriorate the performance of the cell.

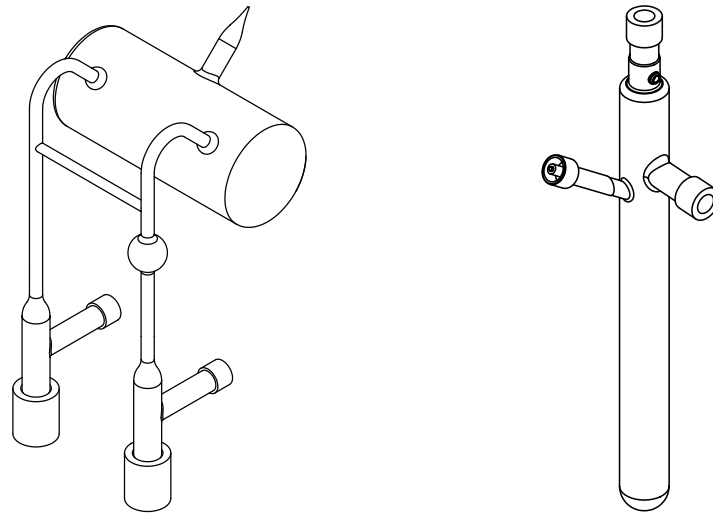


Figure 2: ^{129}Xe optical cell (left) and cold finger (right)

Cold Finger

The cold finger is shown in **Figure 2** and has the following properties:

- Material: Pyrex
- Internal gas volume $\leq 25 \text{ cm}^3$
- Xe collection efficiency $\geq 90\%$ for $F \leq 1.7 \text{ slm}$
- The cold finger contains a gas warming jacket that prevents clogging of the inlet tube and nozzle with solid frozen Xe.
- Capable of holding solid Xe equivalent of 1.0 L of Xe gas at STP.
- Permanent magnet around Xe collection region provides a field $B \geq 2000$ gauss.

Typical Settings

Use the following typical settings when beginning the polarization process:

Table 2: Polarization settings

Item	Value
Xe Mix Pressure	65-75 psig
Purge Gas Pressure	5-20 psig
Oven Air Pressure	20-25 psig
Over Air Flow Rate	120-160 SCFH
Cell Temperature	140°-180°C
Cold Finger N ₂ Pressure	5-10 psig
Cold Finger Jacket Flow	4 SCFH

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5

Installation



The polarizer should be installed in a suitable location, away from high traffic areas and close to the required utilities. (See “Utility Requirements” in Chapter 3.)

Security measures to prevent unauthorized use, theft, or damage to the unit are highly recommended (a locked room, proximity alarm, or other measures).

Before you install any component of the polarizer, please do the following:

1. Verify that the shock or tip sensors on the packaging have not been activated due to rough handling during shipment.
2. Carefully remove the external packaging from the polarizer. Store the packaging for future use in case you must transport the polarizer by vehicle at a later date.
3. Examine the polarizer for visible signs of damage during shipment and verify that all items on the packing list arrived intact.

Plumbing and Electrical Connections

1. Position the polarizer in its chosen operating location, and lock the wheels by pushing down the silver tabs.
 2. Securely mount the Xenon mixture tank in a tank mount close to the polarizer and the UHP purge gas source.
 3. Remove the front and rear panels of the polarizer. Make sure the vacuum pump and laser are still properly secured.
 4. Verify that all AC components are properly connected to the central grounding bar with grounding straps.
 5. Check the vacuum pump oil level (see Chapter 11). Replace the polarizer front and rear panels.
-

6. Connect the main power cable from the unit to the 208 VAC, three-phase local supply with isolation device and supply power.
7. Connect the fitting on the regulator manifold to the Xenon mixture tank. Hand-tighten the nut.

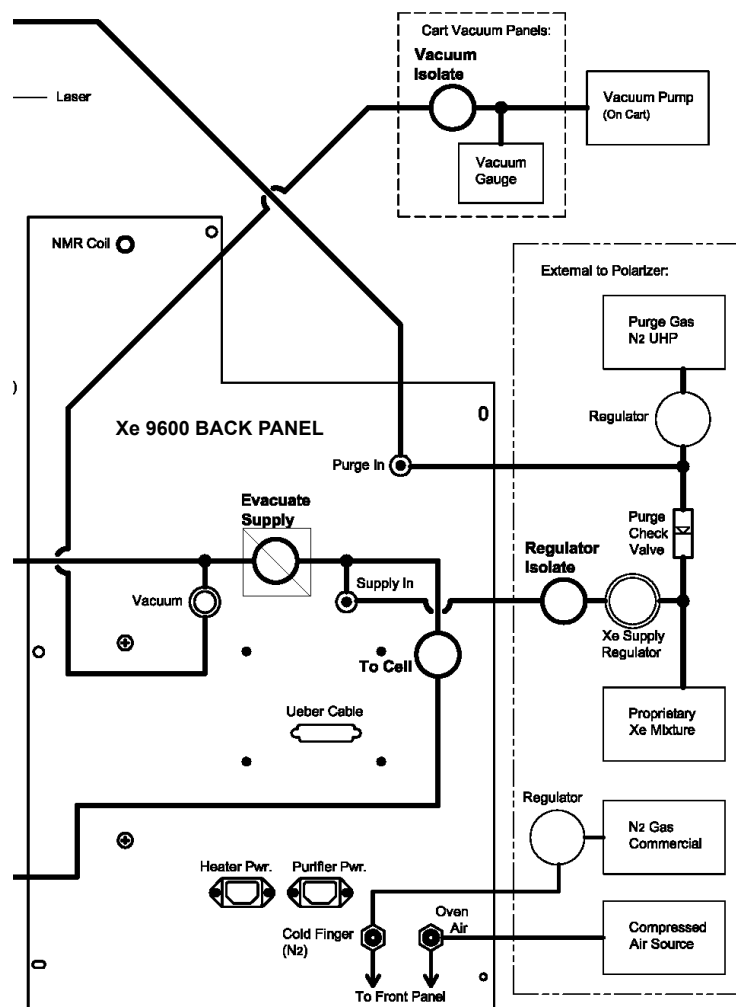


Figure 3: Xenon polarizer rear panel

8. Connect the UHP purge gas at 10 psig to the regulator manifold at the purge gas inlet. The inlet is located at other end of the check valve on the tee between the tank connection and the regulator body. Use 1/4" OD OFHC copper tubing cleaned for O₂ service for the connection if possible. Bend and cut the tubing as needed for the purge gas line to meet all local safety requirements. Tighten the compression fitting on the check valve 1 1/4 turns past finger tight.

9. Using 1/4" OD OFHC copper tubing cleaned for O₂ service, connect the high purity purge gas to the female quick connector on the **Purge In** port on the back of the cartridge – see **Figure 3**. Tighten the compression fittings on the quick connector body 1 1/4 turns past finger tight.
10. Using 1/4" OD OFHC copper tubing cleaned for O₂ service, connect the outlet of the regulator manifold to the female quick connector on the **Supply In** port on the back of the cartridge – see **Figure 3**. Tighten the compression fittings on the quick connector body and the regulator manifold outlet 1 1/4 turns past finger tight.
11. Connect the low pressure dry nitrogen and compressed air (20 psig) lines to the female connectors on the corresponding inlets on the back of the cartridge. Use 1/4" ID tubing rated for at least 50 psig continuous service for the compressed air and nitrogen.
12. Activate the compressed air and dry nitrogen supplies, and verify that you can achieve flow rates of at least 10 SCFH of dry nitrogen and 180 SCFH of compressed air on the gas flow controllers on the cartridge front panel. After you have verified this, shut off the gas supplies.
13. Verify that all valves on the unit (in front and on the back) are closed snugly (except for **Flow Control** valve). Confirm that the quick connector for the vacuum line is plugged securely into the mating connector of the **Vacuum Inlet** port on the back of the cartridge – see **Figure 3**.



Never over-tighten the **Flow Control** valve as it can be damaged.

14. Turn on the unit by momentarily rotating the switch at the main power control panel to the "Start" position (the switch will spring back to the On position where it should be left for normal operation.) If the vacuum pump does not start automatically, remove the front transparent panel and turn on the pump using the power switch located on top towards the back.



If the unit does not power up, make sure the "Emergency Off" button on the main power control panel is not pushed in. (Release the button by twisting clockwise until it pops out.)

15. Open the **Vacuum Isolate** valve, and verify that the pressure reading on the digital vacuum gauge begins to drop.
 16. Make sure the Xenon mixture tank valve is fully closed. Loosen the regulator connection to the mixture tank 1 turn.
 17. Open the high purity purge gas supply and set it to 10 psig. Adjust the **Purge Gas Shut-Off** valve (or the main UHP purge gas supply valve) until the purge gas can barely be heard and felt flowing from the connection to the Xenon mixture tank.
-

18. Allow the purge gas to flow through the tank connection for one minute, and then fully tighten the connection to the Xenon mixture tank.
19. Close the **Purge Gas Shut-Off** valve on the regulator manifold (or the main UHP purge gas supply valve) and open the **Evacuate Supply** valve on the back of the cartridge. Wait for the pressure to drop below 100 mTorr on the vacuum gauge.
20. Close the **Evacuate Supply** valve and open the **Purge Gas Shut-Off** valve (or the main UHP purge gas supply valve). Leave it open for at least 10 seconds.
21. Repeat steps 19 and 20 two more times.
22. Open the **To Cell** valve on the back of the cartridge.
23. If the polarizer is shipped with a removable hard drive, install the enclosure in the computer and lock it in place with the supplied key. (The hard drive will not receive power unless it is locked in place.)
24. Turn on the UPS (if applicable), computer, and monitor.

Cold Finger Installation

See Chapter 4 for a description of the cold finger.

1. Unpack the cold finger and make sure it is intact.
2. Verify that the small tubing for the warming jacket goes to the bottom of the nozzle.
3. Place the cold finger in the cold finger support platform.
4. Attach the cold finger to the cartridge according to the instructions provided in Chapter 8, "Attaching the Cold Finger".

Optical Cell Installation

See Chapter 4 for a description of the optical cell.



The optical cell contains up to 1 gram of rubidium metal. *Rubidium can ignite spontaneously and explosively if exposed to water or air.*

See the MSDS for rubidium (Appendix E) for more information.

1. Put on protective safety eyewear.
 2. Remove the optical cell from the protective packaging.
-

3. Check the rubidium in the main body of the cell for signs of visible contamination. Pure rubidium resembles silver. Signs of contamination include:
 - yellow or brown edges
 - gold color
 - a liquid state at room temperature
 - a white coating on the inside of the main body

Note: A blue or purple hue on the rubidium or cell walls is fine.
4. Install the cell in the cartridge according to the instructions provided in Chapter 11, "Replacing the Optical Cell", beginning with step 10.

Finishing the Installation

Before operating the polarizer, verify the beam path protective housing integrity as described in Chapter 11, "Protective Housing Maintenance".

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Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.

6

Operating the Polarizer

To operate the polarizer, power up the polarizer and the laser. The following sections explain how to operate each component.

Polarean recommends that the polarizer be powered up only once, upon installation, and then left in stand-by mode in between uses.

Note: To complete some of the described procedures, certain tasks need to be performed in software applications. For more information on software applications, see Appendix A.

Powering Up the Polarizer

To power up the polarizer, complete the following steps:

1. Verify the following:
 - the polarizer has proper input power.
 - the compressed air source is attached to the oven air inlet and the pressure is set to 20–25 psig.
 - the source of commercial grade nitrogen at 5–10 psig is attached to the cold finger N₂ inlet.
 - the source of UHP purge gas (99.999% nitrogen or helium) is attached to the xenon manifold and the pressure is set to 20–25 psig.
 - the Xe mixture supply is attached.
 2. Turn the switch on the Power Control Panel to “Start”.
 3. Turn the Temperature-Interlock Unit power (red switch) to “On”.
 4. Verify that the pressure and mass flow readouts on the cartridge are both on.
-

5. Enable power to the laser.
6. Turn on the power supply for the Helmholtz coils by flipping the toggle switch to “On”.
7. Turn on the computer.

Turning the Laser On and Off

Turning On the Laser



Please be aware of the danger of exposure to hazardous visible and invisible laser radiation.



Only trained operators should have access to laser control keys.

1. Secure curtains, doors, interlocks, or laser-attenuating partitions; and illuminate any warning signs.
2. Verify that all laser shielding is in place and that the cryogen access door is closed.
3. Turn on the airflow to the oven to 120–160 SCFH using the airflow indicator on the front of the cartridge.
4. Press “Reset” on the T/I Unit front panel. Verify that both indicator LEDs read normal (green).
5. Power up, arm and activate the laser. See Appendix A for more information on the specific laser control software. Start the Polarimetry software application (double-click the “Polarimetry icon” on the desktop). If the Polarimetry software was already running, exit and re-start the application. (This is necessary to ensure a correct laser light transmission baseline reading).
6. Verify that the Pulse Parameters configuration is set to “Xenon Polarizer”. If it is not correct, click the text and choose the correct setting from the drop-down menu.
7. Verify that the “% Transmission” reading in the bottom left corner is visible and is less than 105%. If it is greater than 105%, exit and re-start Polarimetry.
8. Enable temperature controller output to the air heater (the switch to the left of the oven temperature controller on the T/I unit). Verify that the cell temperature begins to rise on the oven temperature display within 2 minutes.

Turning Off the Laser

1. Turn off the laser according to the directions included with the laser.



In case of an emergency turn off the illuminated toggle switch on the Spectra Physics Integra front panel, or shut down the polarizer using the main emergency power switch.

2. Turn the laser control key to “Off” or “Disable” position.
3. Remove the key and store properly.

Leaving the Polarizer in Stand-By Mode

Polarean recommends the polarizer remain in stand-by mode between uses.

1. Turn the heat switch on the T/I unit to “Off” position.
2. If the laser is emitting, turn it off.
3. Ensure that the cell temperature is decreasing on the T/I control panel.
4. Return all valves to the pre-polarization configuration as shown in **Figure 5**.
5. Turn off the vacuum pump and close the *Vacuum Isolate* valve.

Shutting Down the Polarizer

After putting the polarizer in stand-by mode, perform the following steps:

1. Exit all programs running on the computer.
Note: If using IntegraSoft software, select "Close Port" from the drop-down menu before closing the application.
2. Shut down the computer via the start menu. Once the computer is powered off, close the lid and slide the drawer all the way into the unit.
3. If the polarizer is not going to be operated for an extended period of time (e.g. several weeks), close the glass inlet and outlet valves of the cell (see procedures in Chapter 11)
4. Turn off the T/I Unit and the Helmholtz coil power supply.
5. Close the *Cell Isolate*, *Cell Out*, and *Flow Isolate* valves.
6. Turn off power to the unit at the power control panel switch by rotating the switch to the “Off” position.

Revision History

Date	Change	By whom
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7

Polarizing Xenon

There are four steps to polarizing Xenon:

- Prepare the polarizer.
- Activate the laser while flowing xenon.
- Collect the polarized Xenon.
- Thaw the collected Xenon.

Note: For some of these procedures, tasks are performed in software applications. For more information on software applications, see Appendix A.

Initializing the Xenon Collection

1. Verify the Helmholtz coil power supply is on.
2. Check that all valves are in the proper initial configuration. (See **Figure 4.**)
Note: A complete plumbing layout appears in Chapter 11.
3. If the optical cell glass inlet and outlet valves on the optical cell were closed due to extended shutdown since the last time the polarizer was run, open both valves by following the instructions provided in Chapter 11, “Pressurizing the Optical Cell”.

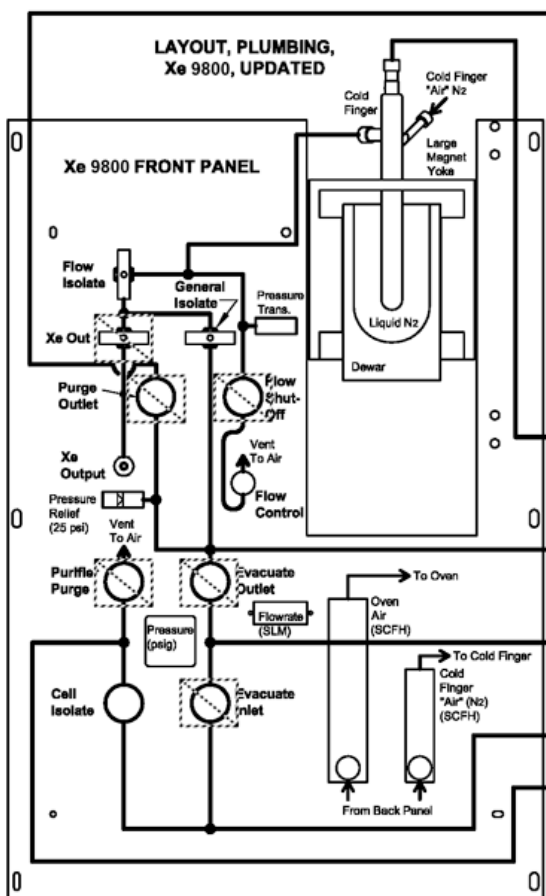


Figure 4: Xenon 9800 polarizer front panel

4. Turn on the vacuum pump, open the *Vacuum Isolate* valve (if not already open) and verify that the outlet manifold pressure is 100 mTorr or less on the vacuum gauge.
5. Evacuate the outlet manifold up to *Cell Out* valve by opening the *Evacuate Outlet* valve until the pressure is 100 mTorr or less.
6. Close the *Evacuate Outlet* valve, and then open the *Purge Outlet* valve for approximately 2 seconds and close it.
7. Open the *Evacuate Outlet* valve and wait for the pressure to fall below 100 mTorr.
8. With the *Purge Outlet* valve closed, *Evacuate Outlet* valve open and pressure below 100 mTorr, close the *Vacuum Isolate* valve and ensure that the pressure does not rise more than 1-2 mTorr per second. If it does, open the *Vacuum Isolate* valve and repeat steps 6 and 7 until the system is thoroughly evacuated of impurities before proceeding.
9. Open the *Vacuum Isolate* valve, close the *Evacuate Outlet* valve and then open the *Purge Outlet* valve for approximately 2 seconds and close it.

10. Close the *Flow Isolate* valve to isolate the cold finger.
11. Place the empty liquid nitrogen dewar directly below the cold finger, then do the following:
 - a. Raise the dewar around the cold finger.
 - b. Insert the shelf below the dewar to hold the dewar in position.
 - c. Verify that the liquid nitrogen inlet, located on top of the housing, is inserted into the dewar.
 - d. Close the cryogen access door.
12. Open the Xenon mixture supply tank and set the regulator on the Xenon mixture tank to 75 psig.
13. *Slightly* open the *Cell Out* valve to pressurize the outlet manifold at a rate of 1 to 2 psi per second. After the pressure reaches 75 psig, open the *Cell Out* valve fully.
14. Turn on the oven airflow and use the float-type flow meter to adjust the airflow to 120–160 SCFH.
15. Press “Reset” on the interlock.



Both indicators' LEDs should be green before proceeding.

16. Flip the audio alarm toggle switch “On” if you are using a door or curtain interlock connected to the polarizer.
17. Set a slight xenon gas flow rate by performing the following steps:
 - a. Close the *Flow Control* valve with *only* two fingers until resistance is barely felt (do not over tighten).
 - b. Open the *Flow Control* valve one-half turn.
 - c. Open the *Flow Shut-off* valve 1 turn.
 - d. Set the flow rate to ~0.1 SLM using the *Flow Control* valve.



Never over-tighten the *Flow Control* valve as it can be damaged.

18. Power up the laser and then start the Polarimetry software as described in Chapter 6, “Turning the Laser On and Off”.
19. Set the oven controller temperature to the final temperature from the last operation of the polarizer (if it is a new cell or the polarizer has not been run in more than 2 weeks, set it initially to 150°C) and enable power to the air

heater (the switch to the left of the oven temperature controller on the T/I unit). Verify that the cell temperature begins to rise on the oven temperature display within 2 minutes.

Collecting Hyperpolarized Xenon



Liquid nitrogen can cause severe frostbite if it comes in contact with skin, eyes, or clothing. Always use appropriate caution and protective gear.

1. Start the nitrogen flow to the cold finger warming jacket by ensuring the supply gas is open and adjusting the flow to 4 SCFH (read at the center of the ball on the small float-type flow meter).
2. Adjust the oven controller temperature to maintain a <50% transmission for broad lasers (>2 nm FWHM) or <15% transmission for narrowed lasers. The temperature should be as low as possible while still maintaining transmission below this threshold.
3. Use the **Flow Control** valve to adjust the Xenon mixture flow to the suggested rate using the following formula*:

$$100 \times \text{Desired Volume [L]} / \text{Time of Run [min]} = \text{Flow Rate [L/Min]}$$

* for 1% Xenon mixture.



Xenon collection times in excess of 60 minutes typically result in lower overall polarization due to solid ^{129}Xe relaxation effects. However, high flow rates can also reduce polarization because of decreased optical spin pumping time within the cell, therefore it is important to strike a sensible balance between these two effects. A reasonable setup for 1 L of gas would be to accumulate for 60 minutes at ~1.67 slm. Please refer to **Figure 1** (Chapter 4) for a set of representative polarizer performance curves.

4. Place the funnel in the liquid nitrogen inlet fitting, then perform the following:
 - a. Pour liquid nitrogen into the funnel to begin accumulation.
 - b. After the rapid nitrogen boil-off stops, fill the dewar to the top.



This is the start time for Xenon accumulation.

- c. If desired, record this start time on a data sheet or else start a timer for the accumulation to ensure collecting accurate volumes.
5. Adjust the flow controller as needed to maintain the desired flow rate.
 6. Adjust the oven controller temperature as needed to maintain the laser transmission just below 50% for broad lasers or 15% for narrowed lasers.

7. Maintain the liquid nitrogen level in the dewar (typically by topping it off every 10 minutes).
8. Check the system at least every 10 minutes during accumulation and adjust the **Flow Control** valve and oven controller temperature to maintain the desired flow rate and laser transmission just below the appropriate threshold. Fill the thawing vessel $\frac{3}{4}$ full with room temperature water (vessel will be needed when the collected Xenon is thawed).
9. Attach a collection container (e.g. an inflatable plastic bag) to the Xe outlet.
10. Evacuate the container by opening the **Evacuate Outlet**, **General Isolate** and **Xe Out** valves and letting the vacuum reach 100 mTorr or below.
11. Purge the collection container with purge gas by closing the **Evacuate Outlet** valve, then opening the **Purge Outlet** valve until the bag is $\frac{3}{4}$ full, if the collection container is flexible, or until the pressure reading is 1 psig in a rigid container.



The pressure of the purge gas is sufficient to burst a bag if overfilled. Use caution when filling bags.

12. Repeat this step several times throughout the accumulation to prepare the collection container for dispensing.

Terminating the Collection

Once the appropriate time has elapsed for collection of the desired volume of Xenon according to the formula in the section above:

1. Increase the flow to the cold finger warming jacket to slightly above 10 SCFH.
2. Close the **Cell Out** valve to stop flow out of the cell. Pressure in the outlet manifold will begin to drop.
3. After pressure drops down to 5–10 psig, close the **Flow Shut-off** valve.
4. Evacuate the residual gas pressure in the outlet manifold by opening the **Evacuate Outlet**, **General Isolate**, and **Flow Isolate** valves.
5. When pressure reaches approximately –14 psig, close the **Flow Isolate** valve. It is not necessary to pull a complete vacuum.
6. Turn off the laser.
7. Turn off the Heat switch on the T/I unit.

Note: If accumulating an additional batch, the laser and heat should be left “On”, otherwise follow “Resetting the Polarizer” procedures.

Thawing Frozen Xenon



Sublimation of any accumulated solid Xenon occurs rapidly once the cold finger is removed from the liquid nitrogen. As the Xenon is returned to the gaseous state, the pressure will build rapidly in the cold finger. Pressure should be monitored carefully during the entire thawing process and valves opened at specific points specified in the procedure. This is why it is greatly encouraged for all operators to ***memorize and practice*** the thawing procedure prior to performing it with accumulated Xenon in the cold finger. Wearing eye protection is recommended during the thawing process.

1. Verify that the ***Flow Isolate*** valve is closed.
2. Evacuate the container by opening the ***Evacuate Outlet***, ***General Isolate***, and ***Xe Out*** valves and letting the vacuum reach 100 mTorr or below.
3. Close the ***Evacuate Outlet*** valve after the vacuum has fallen below 100 mTorr.
4. Close the ***General Isolate*** valve.



Steps 5–8 must be performed with *precise* timing. The procedure and all steps must be read, understood and practiced completely before attempting to perform them.



Failure to perform these steps with precise timing could result in over-pressurization of the system and possible explosion causing operator injury and polarizer damage, and at the very least, low polarization!

5. Verify once again that the ***Flow Isolate*** valve is closed.
6. Support the dewar, and slide its support shelf out from under it.
7. Lower the liquid nitrogen dewar and set it aside.
8. Place left hand on the ***Flow Isolate*** valve while holding the thawing vessel containing room temperature water in the right hand. Now, complete the following steps:
 - a. Lift the thawing vessel containing room temperature water around the cold finger as far as it will go.
 - b. Swish the thawing vessel side-to-side for better heat transfer.
 - c. Monitor the pressure continuously until it reaches 45 psig (this will occur approximately 6-10 seconds after immersion in water).
 - d. Quickly open the ***Flow Isolate*** valve until the pressure falls and then close it immediately. Repeat steps b–c as the Xenon continues to thaw. By keeping the cold finger pressure in the range of 40-60 psig during the thaw, frozen Xenon will spend less time as a warm solid and polarization

is better preserved. It is important to keep pressure high but not to let pressure rise substantially above 60 psig.

- e. When the pressure stops increasing, fully open the *Flow Isolate* valve to extract any remaining Xenon in the cold finger.
 - f. Close the valve to the collection container or close the pinch clamp on the bag tubing.
9. Close the *Xe Out* valve, detach the collection container and place it on the *Polarization Measurement Station* before taking it to the application site.
 10. Turn off the flow to the cold finger warming jacket.
 11. Open the *General Isolate* valve for the next use.

Preparing a Consecutive Batch

Note: The following steps are only for producing an additional batch of Xenon. Otherwise proceed to "Resetting the Polarizer."

It is not necessary to start from the cold state for an additional batch once the polarizer is warmed up.

1. At the end of an accumulation, ensure that:
 - a) The laser is left on.
 - b) The oven temperature control is left on.
 - c) Air flow to the oven is left on.
 - d) Air flow to the cold finger warming jacket is returned to 4 SCFH.
 - e) The **Flow Isolate** valve is closed.
 - f) The liquid nitrogen dewar is empty.
 2. Open the **Cell Out** valve very slowly, so that the pressure only rises 1 psig per second. *It is important that a rush of gas does not leave the cell while it is hot, or Rb may move within the cell.*
 3. Open the **Flow Shut-Off** valve. Allow the oven temperature and flow rates to stabilize, and adjust them accordingly to return to standard operating settings.
 4. Raise the empty liquid nitrogen dewar back into place.
 5. Resume "Collecting Hyperpolarized Xenon" at step 5, and continue to accumulate a batch as described in previous sections.
-

Resetting the Polarizer

Reset the polarizer to return it to an idle, stand-by mode.

1. (Optional) After cell temperature drops below 60°C, turn off the flow of compressed air to the oven.
2. (Optional) Turn the laser control key on the laser control panel to the “Off” position.
3. Close the Xenon mixture supply tank.
4. Turn off the vacuum pump.

The following components can remain powered up for future runs:

- Helmholtz power supply
 - Temperature-Interlock Unit
 - Computer
-

Revision History

Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.

8

Cold Finger Operations

To remove rubidium buildup, remove and rinse the cold finger with distilled water after approximately 20 runs. Dry the cold finger by flowing commercial grade nitrogen or compressed air through it.

Detaching the Cold Finger

1. Verify that the oven temperature is below 40° C.
2. Put on protective eyewear.
3. Open the cryogen access door.
4. Verify that the *Cell Out* and *Flow Isolate* valves on the cartridge are closed.
5. If necessary, open the *Flow Shut-Off* and *Flow Control* valves to depressurize the cold finger to below 15 psig.
6. Loosen the cold finger inlet and outlet compression fittings fully.
7. Detach the cold finger warming jacket Luer-lock connection.
8. Rotate the cold finger away from the horizontal connection, then pull down to detach the vertical connection.
9. Carefully slide the cold finger out of place, while avoiding any unnecessary stress on its body against the magnet yoke.

Attaching the Cold Finger

1. Put on protective eyewear.
 2. Open the cryogen access door.
-

3. Carefully place the cold finger in the cold finger platform, while avoiding any unnecessary stress on its body against the magnet yoke
4. Rotate the cold finger platform through the cryogen door until the o-ring on the cold finger horizontal connection contacts the glass threads.
5. Put the end of the *Cell Out* valve tube into the top connection on the cold finger.
6. Attach cold finger warming jacket Luer-lock connection.
7. Make sure the o-ring for the compression fitting on the top of the cold finger is in the groove in the tubing. Tighten the compression fittings to the cold finger inlet and outlet ports finger tight.
8. With the vacuum on, open the *Evacuate Outlet*, *General Isolate*, and *Flow Isolate* valves to evacuate the cold finger.
9. Close the *Evacuate Outlet* valve, and open the *Purge Outlet* valve for 2 seconds, then close again.
10. Open the *Evacuate Outlet* valve, and allow the vacuum to fall as far as possible. Continue to vacuum for 5 minutes.
11. Repeat steps 12–13 twice more to remove adsorbed water from the interior surface of the cold finger. Close the *Evacuate Outlet* valve, and open the *Purge Outlet* valve for 2 seconds to pressurize the cold finger, then close it again.

Revision History

Date	Change	By whom
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9

The Temperature-Interlock Unit

The Temperature-Interlock (T/I) Unit is installed in the Xenon Polarizer for safety and convenience. It performs the following functions:

- The internal circuitry controls the temperature of the cell by controlling the input power to a forced air heater.
- It provides regulated 12VDC and 24VDC power to the mass flow meter and digital pressure transducer in the cartridge.
- It disables laser emission if the optional external interlock circuit is tripped.

An oven airflow switch stops laser emission and turns off the oven air heater when the oven airflow drops below a safe level (~110 SCFH). To reset the oven air interlock so that you can turn on the laser and oven air heater, you must turn the flow rate above 130 SCFH briefly. After this, you can set the flow to the desired rate.

If either safety interlock is tripped (resulting in a Fault condition), the following events occur:

- The laser emission is disabled.
- An amber light on the front panel of the T/I Unit indicates which circuit has been interrupted.
- The hour timer begins counting the amount of time elapsed since the interlock has been tripped.
- The alarm will sound (if the alarm switch is in the "On" position).

To turn off the alarm, flip the Alarm switch to "Off". Then hit the "Reset" button (to the right of the Alarm switch) to reset the interlocks and re-initiate the laser emission. (Merely switching the Alarm to "Off" does not reset the interlock and does not enable you to use the laser or heater again.)

If power to the T/I Unit turns off, both switches are automatically set to the tripped position. After a power-down or power failure, you must hit the "Reset" button on the front panel before using the polarizer again.



If at any time the Emission light on the laser control panel is on with either of the interlocks in the Fault condition, shut down the polarizer *immediately*, and contact a Polarean service representative.

Controlling Cell Temperature

You can increase cell temperature by the forced air heater only when the air interlock is in the Normal (not Fault) state.

1. Turn the Heat switch to the "On" position.
2. To change the cell temperature, adjust the set point using the up and down arrows on the front of the temperature controller.

Refer to the temperature controller manual for instructions on additional features such as auto-tuning and changing the input control type.

Connecting an External Interlock (Optional)

There is a port on the back of the T/I Unit where you may connect an external interlock or warning sign. If no external interlock is used, the port should be shorted with the supplied jumper.

The port accepts a DB-9 connector and supplies an open circuit potential difference of 5VDC between pins 1 and 6 in the connector. While pins 1 and 6 in the connector are shorted together, the External Interlock can be reset to the normal condition using the Reset button.

The External Interlock will revert to the fault condition if the connection is opened or if power to the T/I Unit is lost.



Never connect a jumper to the Air Interlock port on the back of the T/I Unit. Cell explosion, forced air heater destruction, fire, or severe damage to the polarizer could result.

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10

Cleaning and Transporting the Polarizer

Cleaning the Polarizer

During normal operation, the polarizer does not require any routine cleaning. The surface panels can be cleaned with a soft cloth moistened only with water if they become dirty.



Never use any solvents or non-aqueous solutions on the polarizer as the paint and printed lettering can be damaged. Do not spray any liquids directly on the polarizer. Do not clean the front of the rack-mount components. Water penetrating through the panel cutouts can damage the components.

Moving the Polarizer

It is safe to roll the polarizer across a smooth interior floor surface. For transport outside or with a vehicle, a cushioned skid or pallet must be provided. The procedure for loading onto the skid or pallet will vary and the skid or pallet manufacturer should be consulted.

Whenever the polarizer is transported by vehicle, a 5 G-shock indicator should be affixed to the packaging to indicate rough handling.



The polarizer should never be moved while the optical cell and/or cold finger are pressurized above 14 psig. The glassware could explode resulting in serious injury and damage to the unit. Make sure all gas and electrical lines have been disconnected from the unit before moving.

Preparing the Polarizer for Moving

1. Depressurize the optical cell. (See Chapter 11 for this procedure.)
2. If the polarizer will be transported by vehicle, the optical cell must be removed and packaged separately. Refer to steps 1–9 of “Replacing the Optical Cell” in Chapter 11.
3. Detach the cold finger (see Chapter 8) and remove it along with the dewar from the cryogenic enclosure. Safely package the cold finger and dewar separately.
4. Shut down the polarizer (see the following section).
5. Close all valves on the front and rear panels.



Never over-tighten the *Flow Control* valve as it can be damaged.

6. Remove the compressed air and dry nitrogen inlet lines from the unit by pulling the sleeves on the connectors back until the connectors separate.
7. Detach the Xenon mixture supply and purge gas lines from the back of the cartridge by pushing the flange on the bodies of the quick connectors towards the cartridge until they click.
8. Detach the electrical connection from the wall plug and store safely.
9. Verify that the vacuum pump is securely affixed to the floor of the unit and the laser is properly secured on the rack.
10. Remove any network or external interlock connections from the polarizer and replace all panels.
11. Unlock the wheels by pulling up on the silver tabs above each until they release.
12. Verify the beam path protective housing integrity (see Chapter 11) before you run the polarizer again.

Shutting Down the Polarizer

1. Exit all programs running on the computer.
 2. Shut down the computer and slide the drawer all the way into the unit.
 3. Turn off the T/I Unit and the Helmholtz coil power supply.
 4. Close the *Cell Isolate*, *Cell Out*, and *Flow Isolate* valves.
 5. Turn off power to the unit at the power control panel switch by rotating the switch to the “Off” position.
-

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11

Maintenance Procedures

The following sections describe how to complete standard maintenance procedures on a Xenon polarizer. Although a trained user may conduct tasks, it is recommended that a Polarean service representative perform the outlined procedures.



Always wear protective eyewear when performing maintenance procedures.



Polarean makes no guarantee of the safety or laser housing integrity of the equipment if unauthorized personnel perform these maintenance procedures.

Upon request, Polarean can loan to the customer site all extra equipment needed for performing these procedures (including the infrared viewer and the laser power meter).

Maintenance Schedule

Table 3: Maintenance Schedule

Maintenance Item	Interval	Rationale	Procedure
Verify that all protective housing is in place	Before each run	Safety	Page 4
Verify beam path protective housing integrity	Quarterly or after each service event	Safety	Page 4
Change o-ring gaskets in protective housing	Annually	Safety	Page 5
Check oil level/condition in vacuum pump	Weekly	Life, Performance	Page 6
Change oil in vacuum pump	Semi-Annually	Life, Performance	Page 6
Check optical cell condition	Monthly	Performance	Page 10
Re-activate gas purifier	At every tank change	Performance, Life	Page 13
Clean cold finger	After 20 accumulations or as needed	Performance	Page 14
Perform calibration on the following components: • Outlet Pressure Transducer • Mass Flowmeter	Annually, according to accepted calibration standards	Performance, Safety	Contact Polarean

Plumbing Layout

The complete Xenon polarizer plumbing diagram is shown in **Figure 5**. This diagram should be consulted when performing maintenance tasks.

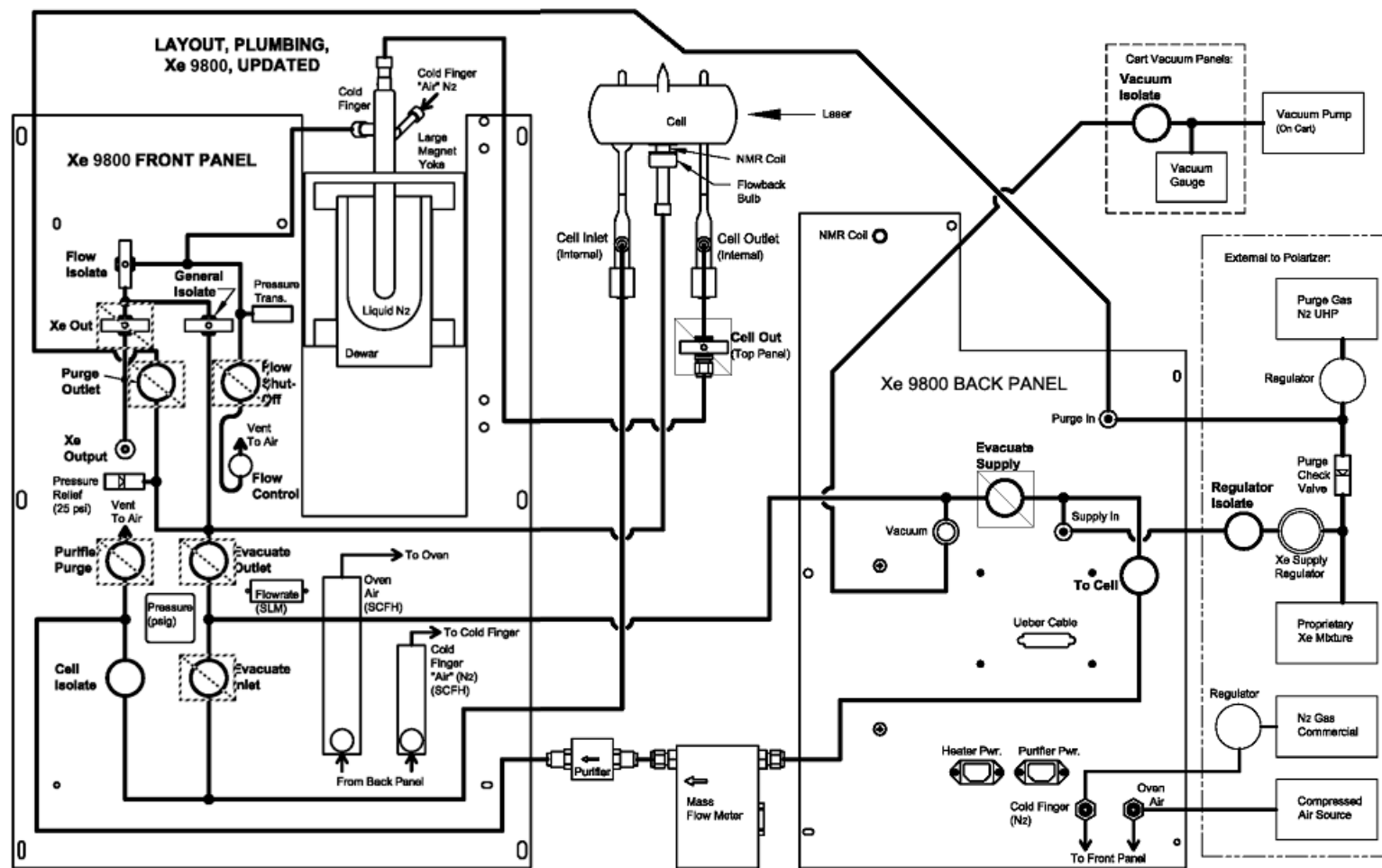


Figure 5: Xenon polarizer plumbing layout

Protective Housing Maintenance



If a calibrated laser power meter is not available for a Radiant Power Emissions Test, the polarizer *must* be considered a Class 4 laser. Consequently, eye protection *must* be worn at all times until a laser power measurement is performed.

Checking for the Presence and Integrity of Protective Housing

It is recommended that this test be performed every day before operating the polarizer.

1. Verify that all pieces of the protective housing (shown in the plumbing layout of **Figure 5**) are in place, including screws and gaskets.
2. If any piece of housing is visibly damaged or its integrity is in question, contact a Polarean service representative.

Verifying Beam Path Protective Housing Integrity

1. Move the product into a room suitable for the use of Class 4 laser products and turn off all other sources of laser energy in the room. Make all necessary electrical, air and external interlock connections to the unit.
2. Clear room of all unnecessary personnel.



Ensure that everyone remaining in the room is trained in laser safety and is wearing laser attenuating (OD>4 at 795 nm) eye protection.

3. Verify that all panels and protective housing pieces are in place on the unit.
4. Set the laser power meter's wavelength detected to 795 nm and set the scale to 1 mW).
5. Turn on the laser.
6. With the infrared viewer set at the widest aperture, check for areas of laser light leakage around the unit's housing sections.
7. Using the laser power meter with a bare detector head, check the power of radiant light at each visible leakage point.
 - a. Ensure the surface of the detector is perpendicular to the radiant light.
 - b. Open all panels and doors designed to be opened during normal operation and check radiant power levels inside.
 - c. Note the maximum reading from all areas checked.

8. Turn off the laser and the laser power meter. Record specifically the areas of leakage with radiant power above 5×10^{-4} watts.

If any radiant emission was above 5×10^{-4} watts, contact a Polarean service representative for proper remedial measures.



Do not operate the polarizer until all sources of excessive emissions have been identified and repaired.

Replacement of Protective Housing Gaskets

Before performing the procedures described in this section, contact a Polarean service representative to check whether any gaskets are installed on your polarizer's protective housing.



This procedure involves removing a portion of the protective housing which encloses the direct laser beam path. While exposure to the direct laser beam is not possible during this procedure, Class 1 laser radiant power output specifications may be exceeded if the housing is not re-installed correctly.

Although a trained user may conduct these tasks, it is recommended that a Polarean service representative perform the outlined procedures. Subsequently, a radiant power output check should be performed immediately upon its completion.

1. Ensure the polarizer has *not* been running for at least 30 minutes. (The oven outer surface should be cool to the touch and the oven temperature measurement below 35°C for this procedure.)
2. Put on laser protective eyewear with $\text{OD} > 4$ at 795 nm.
3. Turn all laser power off, and remove the key(s) from the control panel(s).
4. Remove the four screws on each side that hold the laser tube and backstop to the sides of the oven.
5. Slide the laser tube and backstop away from the oven.
6. Remove the old o-ring gasket.
7. Insert the four screws holding the laser beam enclosure tube to the oven into the holes in the laser tube.
8. Stretch the new $1/8''$ o-ring over the shafts of all four screws.
9. Push the laser tube and backstop back up to the oven and start the four retaining screws on each side in the holes in the sides of the oven.

10. Tighten the mounting screws only enough to compress the o-ring gasket to one-half its original thickness. All four screws on each side should be uniformly snug.
11. Verify that the radiant laser power emission levels around the oven top are within Class 1 laser product limits. (See “Verifying Beam Path Protective Housing Integrity”.)

Vacuum Pump Maintenance

Checking the Oil Level/Condition in the Vacuum Pump

Observe the oil color and level in the sight glass on the front of the vacuum pump:

- If the oil level is below the Min marking, add oil as necessary to keep the level between the Max and Min markings on the housing.
- If the oil looks milky (bubbles are acceptable) or dark amber in color, change the pump oil as described in the following section.
- If there is a puddle of oil in the intake or exhaust traps, or under the body of the vacuum pump, contact a Polarean service representative. (A fine misting is acceptable.)

Changing the Oil in the Vacuum Pump

This procedure should be performed every six months, or as needed.

Note: Use an oil basin (~ 0.5 L) and an oil drain hose with the proper mating connector for the drain plug fitting to collect the old pump oil.

1. Perform the polarizer Shut-down procedure (see Chapter 6).
 2. Close the *Vacuum Isolate* valve on the front panel.
 3. Remove the vacuum pump access panel on the front of the polarizer.
 4. Loosen and remove the QF16 flange clamp between the inlet filter and the vacuum bellows.
 5. Break the connection and place the o-ring on a clean surface.
 6. Remove the drain plug protective cover on the pump housing below the sight glass to expose the drain plug valve. Screw the mating connector from the oil drain hose onto the drain plug fitting until finger tight (oil will start to flow through the hose once the connector is about halfway engaged. Therefore verify the open end of the hose is in a drainage container). Drain the warm oil into the basin until the flow reduces to a slight trickle and the sight glass on the pump is empty.
 7. Disconnect the oil drain hose from the drain plug valve.
-

8. Replace the drain plug protective cover and hand-tighten it.
Note: Please dispose of used oil properly.
9. Remove the oil fill plug from the top of the pump housing above the sight glass.
10. Fill the pump with fresh hydrocarbon or synthetic rotary vane vacuum pump oil (Leybold N62 or equivalent) to a level between the Max and Min marks on the sight glass.
11. Replace the oil fill plug in the top of the pump housing.
12. Re-connect the vacuum hose to the inlet filter. Verify the o-ring is between the filter flange and the vacuum hose flange.
13. Hand-tighten the QF16 flange clamp over the connection.
14. Power up the polarizer and the vacuum pump.
15. Open the *Vacuum Isolate* valve and let the vacuum gauge reading stabilize.
Note: If after 30 minutes, the stabilized reading is >100 mTorr, contact a Polarean service representative.
16. Replace the vacuum pump access panel.

Optical Cell Maintenance

Depressurizing the Optical Cell

1. Put on protective safety eyewear.
 2. Verify the *Cell Out* valve is closed.
 3. Open the *Flow Isolate*, *General Isolate*, and *Evacuate Outlet* valves to evacuate the cold finger.
 4. Allow the pressure in the cold finger to drop below 100 mTorr.
 5. Close the *Flow Isolate* valve.
 6. Close the *Cell Isolate* valve.
 7. Slowly open the *Cell Out* valve and allow the pressure reading to stabilize.
 8. Open the *Flow Shut-Off* and *Flow Control* valves until pressure in outlet manifold reaches 15 psig.
 9. Close the *Flow Shut-Off* and *Cell Out* valves.
-

Pressurizing the Optical Cell

1. Ensure the oven access cover is installed and secured with screws and the cryogenic access door is closed.
2. Ensure the *Cell Out* and *Cell Isolate* valves are closed.
3. Set the outlet pressure on the Xenon mixture regulator to 75 psig.
4. Verify the Xenon mixture tank valve and the *Regulator Isolate* and *To Cell* valves are open.
5. Verify that the cold finger is attached and that the connections to it are finger tight.
6. Open the *Evacuate Outlet*, *General Isolate*, and *Flow Isolate* valves to evacuate the cold finger.
7. When the pressure in the cold finger drops below 100 mTorr, close the *Flow Isolate* valve.
8. Slowly open the *Cell Out* valve to allow the pressure in the cold finger to build at 1 psi per second until the pressure reading is stable.
9. Slowly open the *Cell Isolate* valve to allow the pressure in the cell and cold finger to build at 1 psi per second until the pressure reading is stable.

The polarizer can now be powered up (See “Powering Up the Polarizer” in Chapter 6). If the polarizer will not be used at this time, set the polarizer to stand-by mode by performing the following steps:

- close the *Cell Out* valve
- depressurize the cold finger (Chapter 8)
- open the *Flow Isolate* valve

Checking the Condition of the Optical Cell



This procedure involves removing a portion of the protective housing enclosing the direct laser beam path. While exposure to the direct laser beam is not possible during this procedure, Class 1 laser radiant power output specifications may be exceeded if the housing is not re-installed correctly.

This procedure should be performed only by a person trained to operate in a Class 4 laser environment. Subsequently, a radiant power output check should be performed immediately upon its completion.

1. Make sure the polarizer has *not* been running for at least 30 minutes. (The oven should be cool to the touch for this procedure.)
2. Put on protective eyewear.

3. Turn off the laser power and remove the key.
4. Depressurize the optical cell (as described in “Depressurizing the Optical Cell”).
5. Remove the eight screws holding the top of the oven in place (four on top, four on the back).
6. Gently remove the oven top by lifting it up and back.
7. Check the appearance of the rubidium in the cell. If the rubidium is gold-colored or if it has yellow or black edges, consider replacing the cell (see the following section for this procedure) or consult with a Polarean service representative. Also consider replacing the cell if the interior is significantly coated with a white film.
8. Verify that RTD 1 is firmly affixed to the cell between the cell arms.
9. Make sure the other RTD’s in the oven are not crossing the laser beam path.
10. Replace the oven top and tighten the eight screws holding it in place.
11. Verify that the radiant laser power emission levels around the oven top are within Class 1 laser product limits (as described in “Verifying Beam Path Protective Housing Integrity”).
12. Re-pressurize the optical cell before operating the polarizer (as described in “Pressurizing the Optical Cell”).

Replacing the Optical Cell

This procedure involves removing a portion of the protective housing enclosing the direct laser beam path. While exposure to the direct laser beam is not possible during this procedure, Class 1 laser radiant power output specifications may be exceeded if the housing is not re-installed correctly.



This procedure should be performed only by a person trained to operate in a Class 4 laser environment. Subsequently, a radiant power output check should be performed immediately upon its completion.

After reading the instructions provided in this section, please refer to the video clip enclosed with this operating manual for a demonstration of the steps involved in changing an optical cell.

1. Seal off gas flow to and from the cell by closing the *Cell Isolate* and *Cell Out* valves.
 2. Put on eye protection.
 3. Remove the oven lid.
-

4. Support the valve bodies to keep them from twisting, and then close the glass valve on each of the cell arms until the o-rings contact the glass.
 5. While supporting the cell valves, loosen the Chemthread® connections to each of the cell arms.
 6. Remove the back end of each of the c-shaped cell mounting fixtures inside the oven.
 7. Gently pull the cell toward the back of the oven and detach the RTD from the cell surface.
 8. Slide the cell out the back of the oven. Be careful not to snag the arms in the RTD or NMR wiring.
 9. Remove the new cell from the protective packaging and inspect for signs of visible damage or rubidium contamination (as described in “Checking the Condition of the Optical Cell”).
 10. Gently insert the new cell halfway into the oven by sliding the arms through the foam channels in the bottom of the oven.
 11. Attach the RTD 1 to the front of the cell body just under the pull-off stump. Use strips of 1-2 mil thick, ½” wide Kapton® tape.
 12. Gently slide the cell into the c-shaped mounting fixtures.
 13. Install the back end of the cell mounting fixtures using a hex wrench. Tighten the screws no more than a 1/8 turn past finger tight.
 14. Make a stress-free connection between the Chemthread® connectors on the cell arms and the inlet and outlet tubing. (You may have to bend the tubing slightly and or place the o-ring in a different groove in the tubing to allow for variations in the glass between cells).
 15. Verify that the vacuum pump is on and the *Vacuum Isolate* valve is open. Open the *Evacuate Inlet* valve on the cartridge and allow the pressure to drop below 30 mTorr. Close the *Evacuate Inlet* valve.
 16. Open the *Cell Isolate* valve for five seconds and then close again.
 17. Repeat steps 15 and 16 three more times to eliminate air trapped in the connection to the cell inlet.
 18. Open the *Flow Isolate*, *General Isolate*, *Evacuate Outlet* and *Cell Out* valves. When the pressure drops below 30 mTorr, close the *Evacuate Outlet* valve.
 19. Open the *Purge Outlet* valve for 2 seconds.
 20. Repeat steps 18-19 three more times to eliminate air trapped in the connection to the cell outlet.
-

21. Open the *Evacuate Outlet* valve. When the pressure drops below 30 mTorr, close the *Flow Isolate* valve.
22. While supporting the valve bodies to keep them from twisting, open both glass valves on the new cell arms. (The digital pressure gauge should be reading a slight purge gas pressure at this point.)
23. Replace the oven top and secure with the eight screws.
24. Verify that the radiant laser power emission levels around the oven are within Class 1 laser product limits (as described in “Verifying Beam Path Protective Housing Integrity”).
25. Re-pressurize the optical cell before running (as described in “Pressurizing the Optical Cell”) and close the *Cell Out* valve.

Gas Manifold Maintenance

Replacing the Xenon Mixture Tank

1. Put on protective safety eyewear.
 2. Close the valve on the Xenon mixture supply tank.
 3. Close both *Xenon* and *UHP N₂ Regulator Isolate* valves to maintain pressure in the system.
 4. Verify that the *UHP N₂ Shutoff* valve is open and the UHP nitrogen regulator output is set to 10–20 psi.
 5. Open the connection from the Xenon regulator to the Xenon tank until pressure on regulator begins to bleed down.
 6. Allow pressure to bleed down to 0 on the regulator input gauge. (Purge gas flow will engage to maintain constant outward gas flow and prevent contamination of the regulator.)
 7. Remove the old tank and replace it with the new tank.
 8. Secure the new tank with proper cylinder straps.
 10. Tighten the connection from the regulator to the new tank until it is *1 full turn short of finger-tight*.
 11. Allow purge gas to blow through the gas tank connection for one minute to minimize air contamination of the system.
 12. Use a wrench to finish tightening the connection to the mixture tank.
 13. Open the valve on the new tank.
-

14. Close the tank valve and monitor the regulator inlet pressure gauge for two minutes.

Note: A drop in inlet pressure indicates a leak in the connection to the mixture tank. If this occurs, tighten the connection and repeat steps 12 and 13.

If no leak is apparent, open the valve on the tank fully.

15. Verify that pressure on the outlet side of the regulator is 75 psi on the gauge.
16. Open both *Xenon* and *UHP N₂ Regulator Isolate* valves.

Replacing the UHP Nitrogen Tank

Note: It is important to change the UHP nitrogen cylinder before it becomes completely empty (<150 psig) so that the system may remain pressurized. When the UHP nitrogen cylinder is changed, the connecting hose will be exposed to air. Always try to limit the amount of time the UHP nitrogen hose is exposed to air during the tank replacement process (no more than 1–2 min).

1. Put on protective safety eyewear.
2. Close the *Cell Isolate* valve. This will protect the polarizer and optical cell from air contamination.
3. Close both *Xenon* and *UHP N₂ Regulator Isolate* valves to maintain pressure in the system.
4. Open and close the valve on Xenon mixture tank to pressurize the Xenon line. This will prevent N₂ flow into the Xenon circuit, up to the check valve near the UHP N₂ regulator, and will protect the Xenon side of the circuit from air contamination.
5. Close the valve on the old UHP nitrogen tank.
6. Close the *UHP N₂ Shutoff* valve.
7. Disconnect the hose from the UHP nitrogen tank.
8. Remove the old tank and replace it with the new UHP nitrogen tank.
9. Attach the UHP nitrogen hose to the new tank, tighten the connection using a wrench and open the valve on UHP nitrogen tank.
10. Open the *UHP N₂ HP Vent* valve very slowly until purge gas starts flowing through the orifice. Let the purge gas bleed through the orifice for at least 30 seconds. This is desired because it purges most of the air contamination out of the connecting hose.



NEVER open the *UHP N₂ HP Vent* valve all the way.

11. Close the **UHP N₂ HP Vent** valve.
12. Open the **UHP N₂ Shutoff** valve. The pressure reading on the regulator input gauge should increase reflecting the pressure inside the UHP nitrogen tank.
13. Close the main valve on the UHP nitrogen tank. If the pressure level on the regulator input gauge falls for more than 50 psig in one minute, tighten the connections, open–close the main tank valve and repeat this step. If the leak test passes successfully, fully open the main valve on the UHP nitrogen tank.
14. Set the output of the UHP nitrogen regulator at 20 psig.
15. Slightly open the **UHP N₂ LP Vent** valve until purge gas starts flowing through the orifice. Let the purge gas bleed through the orifice for at least 1 minute. This will flow any room air that may have got into the line back out into the room.
16. Close the **UHP N₂ LP Vent** valve
17. Open both **Xenon** and **UHP N₂ Regulator Isolate** valves.
18. Re-open the **Cell Isolate** valve that was closed in the first step.

Regenerating the Gas Purifier

1. Put on protective safety eyewear.
 2. Verify that all valves are in the default configuration (shown in the plumbing layout in **Figure 5**).
 3. Close the valve on the Xenon mixture tank (but leave the Xenon regulator setting unchanged).
 4. Check that all valves to the 10-psi UHP nitrogen purge gas supply are open.
 5. Close the **Cell Isolate** valve on the front panel.
 6. Open the **Purifier Purge** valve one turn, then close it until the flow rate reads 0.30 L/min on the mass flowmeter readout.
 7. Let the purge gas flow for 10 minutes at this rate.
 8. On the Temperature-Interlock Unit front panel, do the following:
 - a. Verify the Heat switch is Off.
 - d. Lift the cover on the Activate Purifier switch and press the red button until the light comes on.
 - e. Verify the Purifier Control set point is set at 350 °C (press the SEL key to toggle between set point and temperature reading).
-

13. Allow the purifier temperature to reach the set point.



The purifier should be heated at 350 °C for 3 hours with a constant flow of purge gas at 0.30 L/min.

14. After the purifier is regenerated, lift the cover and press the Activate Purifier button until the light goes off.
15. Allow the purifier to cool for 30 minutes with a 0.30 L/min purge gas flow continuing through it.
16. Close the *Purifier Purge* valve.
17. Open the Xenon mixture tank valve.
18. Re-pressurize the optical cell before resuming gas polarization (as described in "Pressurizing the Optical Cell") and open the *Cell Isolate* valve.

Cleaning the Cold Finger

Because the cell is operating at high temperature and under continuous flow, some of the rubidium vapor can leave the cell and condense in the cold finger. This will appear as a white film on the inside walls of the cold finger which can be removed by rinsing the cold finger with distilled water. This procedure should be performed after every 20 accumulations of Xenon in the cold finger or more often if desired.

Rinsing the Cold Finger

1. Remove the cold finger using the instructions in Chapter 8, "Cold Finger Operations".
2. While holding the cold finger upright over a sink or other waste container, fill the cold finger with distilled water.
3. Invert and/or tilt the cold finger to allow the water to drain out.
4. Repeat steps 2 and 3.
5. Try to get as much water as possible out of the interior chambers of the cold finger. You can dry the cold finger by attaching it to a supply of dry pressurized gas such as Helium or Nitrogen until moisture is removed.
6. If there is a white film remaining on the inside walls of the cold finger, it is either contamination from another substance or a slight etching of the glass surface resulting from contact with rubidium. This does not affect the performance of the cold finger or the level of Xenon polarization. Contact a Polarean service representative to obtain a replacement cold finger if desired for aesthetic reasons.

7. Install the cold finger using the instructions in Chapter 8, "Cold Finger Operations".

Revision History

Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.
1/10/2013	UHP N ₂ tank replacement procedures added	Polarean, Inc.
3/14/2013	UHP N ₂ tank replacement procedures revised to reflect the gas handling manifold valve labels	Polarean, Inc.



Using the Computer Applications

There are two software applications the operator uses to operate the Xenon polarizer. Each application is responsible for a different set of tasks:

- *Laser control software* (IntegraSoft) – The operator will use this application to start and stop laser emissions and to check on operating parameters (such as diode temperature).
- *Polarimetry software* (Polarimetry application, version 4.1 or above) – This is a custom application to monitor various aspects of polarizer performance. Periodically during optical pumping, the operator may want to verify the presence of polarized Xenon in the optical cell or need to monitor the amount of laser light being transmitted through it.

Note: Due to the high field strength and proximity of the cold finger to permanent magnets, measurements of the *level* of Xenon polarization in the optical cell are not reliable, even if the system will report them.

The following sections explain how to navigate each application and complete their associated tasks.

The Laser Control Application (IntegraSoft) for Systems with Spectra-Physics Lasers

The IntegraSoft application is developed and maintained by Spectra-Physics (Newport) Corporation, but a copy is included with each polarizer for laser operation and basic functionality is described below.

The IntegraSoft interface supports a single Spectra-Physics Integra laser system, but does not include optical spectral information. This is because the Integra laser system integrated into the polarizer is spectrally narrowed and wavelength controlled by a variable holographic grating (VHG) which is not operator adjustable. If you suspect a laser performance failure, please contact a Polarean service representative.

Starting the Interface

1. Verify that the laser is powered on, that the key is in the "Enable" position and that all of the interlocks show "Normal" (green) on the T/I box.



2. Click the "IntegraSoft" icon on the computer desktop to open the Port Control window, shown in Figure 6. The Port Control window may appear under the main application window but can be brought to the foreground by clicking "Port Control" in the task bar.
3. Verify that Port 1 and 9600 Baud rate are selected and click "OK".

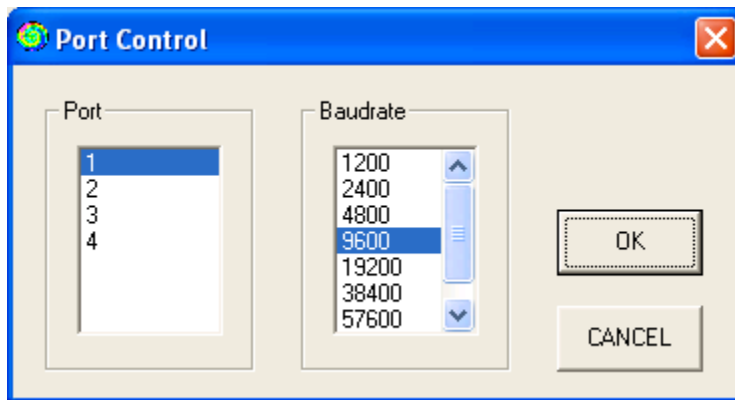


Figure 6: IntegraSoft Port Control window

4. In the IntegraSoft main window, click "Communication" and select "Open Port" from the drop-down menu. The window will display the message "Starting Port..." (see Figure 7)

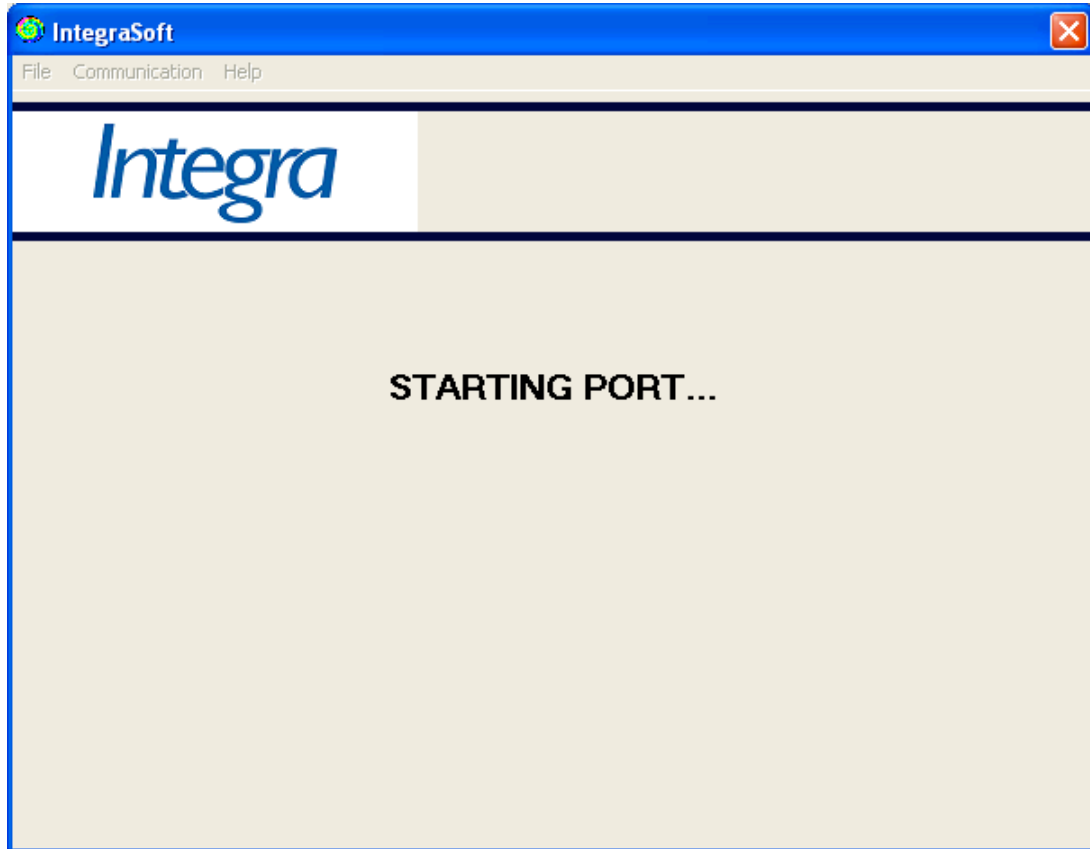


Figure 7: Starting Port... message in IntegraSoft window

5. Messages about communications with the laser will appear in the IntegraSoft application window and disappear automatically as the application starts.
6. Once the application has completed startup and successfully communicated with the Spectra-Physics laser, the window will appear similar to that shown in Figure 8.

Starting Laser Emission

1. To enable and start laser emission, verify that there are no error messages displayed on the LCD panel on the front of the laser then click the On button in the top left corner of the IntegraSoft application window. Laser emission will start after a 5 second delay and Laser Status will change from Off to "EMISSION".
2. The "STBY" button in the application will allow the laser to complete the 5-second delay prior to emission, but without starting the laser emission itself. Five seconds after clicking the "STBY" button, the Laser Status will display as "STANDBY". From this state, emission can be started immediately (without any further delay) by clicking the On button or can be returned to the OFF state with the Off button. The Stand-By state is not used in any procedures for the polarizer but is described here for completeness.

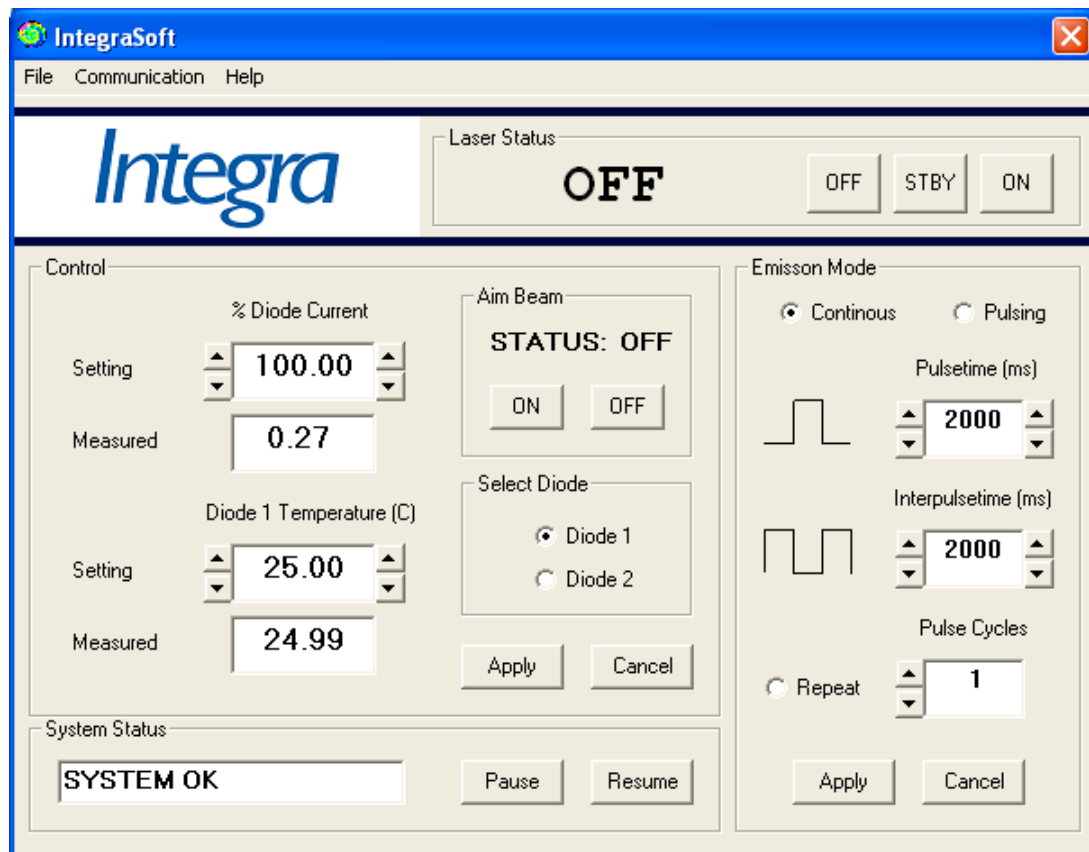


Figure 8: IntegraSoft main window.

3. The Spectra-Physics Integra laser incorporates a variable holographic grating (VHG) for each diode to control the output wavelength. Adjusting the temperature of the diodes WILL NOT adjust the wavelength of the output. The diode temperature settings should not be adjusted without first consulting with Polarean as it may adversely affect polarizer performance. However, real-time diode temperature can be monitored by using the radio buttons under "Select Diode". The "Apply" button does not have to be clicked to switch between the diodes.
4. Emission mode should always be set to "Continuous" (yes, it should be "continuous" but there is a typo in the software interface). Even though it is present in the software interface, the system does not incorporate an aiming beam so the buttons to turn the aiming beam ON and OFF do nothing in reality.
5. Laser output is nominally 50W CW centered on 794.6 nm wavelength. The diode current may be adjusted to any percent of maximum below 100% to adjust the output power, but the VHG may not hold the wavelength at 794.6 nm at diode current levels below 90%. Current for each diode cannot be set independently.

The Polarimetry Application (version 4.1 and above)

The Polarimetry application can be used to both detect the presence of polarized ^{129}Xe in the cell and monitor the fraction of laser light being transmitted through the optical cell during gas production. Keeping % Transmission at prescribed levels by varying cell temperature will optimize the polarization of the gas dispensed. The application contains three different views:

- The Data Acquisition view — allows the completion of all tasks during gas production, such as acquiring NMR data and monitoring laser transmission (see **Figure 10**).
- The Inventory view — displays serial numbers of the polarimetry hardware (see Figure 13).
- The Configuration view — displays specific settings for Hardware Parameters, NMR (Nuclear Magnetic Resonance) Circuits, Pulse Parameters, Alert Tolerances, and Gas Sample Standards (see Figure 14).

The Data Acquisition view opens by default when the polarimetry application is launched. Most actions take place in this view.

Navigating the Data Acquisition View

The Data Acquisition view contains the following features:

- Pulse Parameters drop-down list box — defines the type of gas (Helium or Xenon) and the type of system (Polarizer or Polarimetry Station) being used
- Acquire button — measures and displays the level of polarization of the gas at that exact moment
- Exit button — exits the polarimetry application

Similarly, there are two tabs on the Data Acquisition view.

- Spin-Up/-Down Tab — Allows running a study of the polarization levels over a period of time. (See “Running a T1 Study.”)
 - FID Analysis Tab — Allows revising the gas sample setting values (sample pressure and sample blend) *only* if specifically instructed to in an SOP. (See “Changing Sample Pressure and Blend.”)
-



Figure 9: The polarimetry window-Data Acquisition view

Gas Sample Settings: Pressure, Blend, and Pulse Parameters

Gas Sample Settings describe the gas being used in your system (in a cell or bag). These values are important because they are used in determining the level of polarization in your gas.

The Gas Sample Settings comprise the following:

Pulse Parameters Set – This defines the type of gas (Helium or Xenon) and the type of system (Polarizer or Polarimetry Station) being used. Only Xenon Polarizer and Circuit Test – Low Frequency should be selectable.

Sample Pressure – This is the pressure at which a gas sample is held.

Sample Blend – This is the percentage of a gas sample that contains the isotope of interest for polarization.

A Polarean service technician sets default values upon installation. Values can be edited, but they should *never* be changed unless specifically instructed to in an SOP.



Setting these values incorrectly could lead to invalid measurement of polarization.

Changing Pulse Parameters Set

Use the Pulse Parameters drop-down list box to select what Pulse Parameter set will be in use. To change this selection, click the drop-down control and choose the desired set from the list.

Changing Sample Pressure and Blend

Both the Sample Pressure and Sample Blend settings each have a “Use Default?” check box. If the “Use Default” box is enabled (has an ‘X’ in it), the standard value for that setting will be used.

If the box is cleared (does *not* have an ‘X’), then the setting may have been changed by a user.

Note: These boxes are automatically checked whenever the program is re-started or the selection of Pulse Parameters Set is changed, because the standards values for these settings are re-loaded at such times.

To change the value of either setting, complete the following steps:

1. Click on the “Use Default” check box to clear it.
2. Click in the “number box” and type a new value for the associated setting.

Performing Data Acquisitions

The polarimetry window allows to measure the polarization level in the gas. This is accomplished by clicking the “Acquire Data” button in the polarimetry window.

After clicking “Acquire Data”, the Polarization (%) field displays the level of polarization of the gas at that exact moment. Also, fields on the FID Analysis tab are updated with additional information about the acquired signal.

Notifying the Operator of Uncertainty in Calculated Values

If the calculated value for T_2^* is less than 4 ms or if the calculated value for SNR is less than 2, the background color behind the display of the value (as well as the background behind the % Polarization) will change to orange. This is intended to let the user know that there is less than an 85% certainty in the values being displayed.

Increasing Signal-to-Noise for Low Amplitude Signals

For measuring signals with a low amplitude compared to the background electromagnetic noise, there are some processing algorithms that can be used to help increase the signal visibility: averaging of multiple signal acquisitions and line broadening of the individual measurements. These algorithms can be used either independently or together.

Averaging

This is used to reduce the background noise levels for random and near-random sources of noise (ie not always at the same frequency). The effect of this is only visible in the Frequency domain graph. The FT-processed results of the selected number of acquisitions will be summed and displayed as a single acquisition. Random noise will tend to be reduced in proportion to the square-root of the number of averages selected out while the signal from polarized gas will be increased. To turn on averaging, click the switch marked “Average?” (see Figure 10) in the bottom left corner of the Polarimetry software window (the indicator on the switch should turn bright green) and input the number of averages desired and the time between measurements (seconds). Averaging will be performed every time data is acquired (depending on the number of averages selected, this can greatly increase the time required for data acquisition).



Figure 10: Averaging control interface

To turn off averaging, click the switch marked “Averaging” again (the indicator on the switch should turn dark green or black).

Broadening

This algorithm applies an exponential amplitude decay function to the time-domain signal to reduce levels of coherent or non-random background noise. The value input in the interface determines the value of the exponential or the degree of broadening. Larger values for the exponential give more line broadening. The effect can be seen in both the time and frequency domain graphs (see Figure 12 for an example).

To turn on line broadening, click the switch marked “Broadening?” (see Figure 11) in the bottom middle portion of the Polarimetry software window (the indicator on the switch should turn bright green) and input the degree of line broadening desired in the “A” field (0.01 is a good starting value).

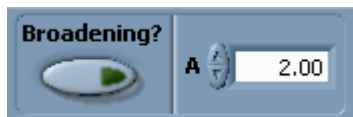


Figure 11: Broadening control interface

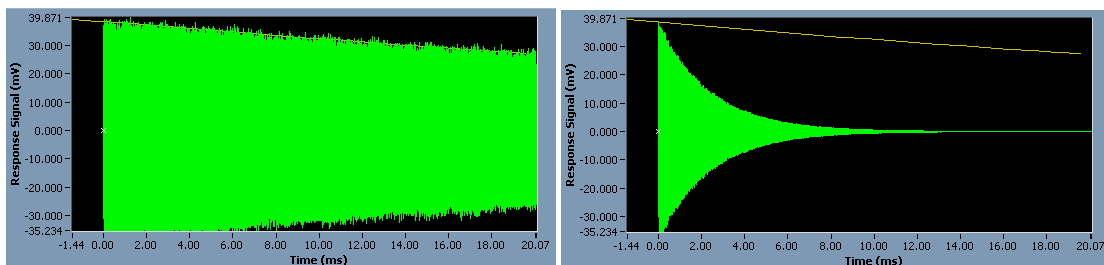


Figure 12: Polarized gas signals without (left) and with (right) broadening applied

Recording the Baseline Transmission

A baseline reading of the photodiode voltage is taken whenever Polarimetry is launched. This value is used as the reference point for 100% transmission. Therefore, for accurate measurements of laser power transmitted through the cell, the Polarimetry application should only be started with the cell temperature below 50 °C. If IntegraSoft is being used for laser control, the Polarimetry application should be launched only after laser emission has started and before the cell reaches a temperature of 50 °C

Viewing Polarimetry Inventory Information

The Inventory view displays information about the polarimetry application itself, both the hardware and the software components.

The information on these views is for viewing purposes only and cannot be changed. A Polarean service representative may request this information, however, if the polarizer experiences technical problems.

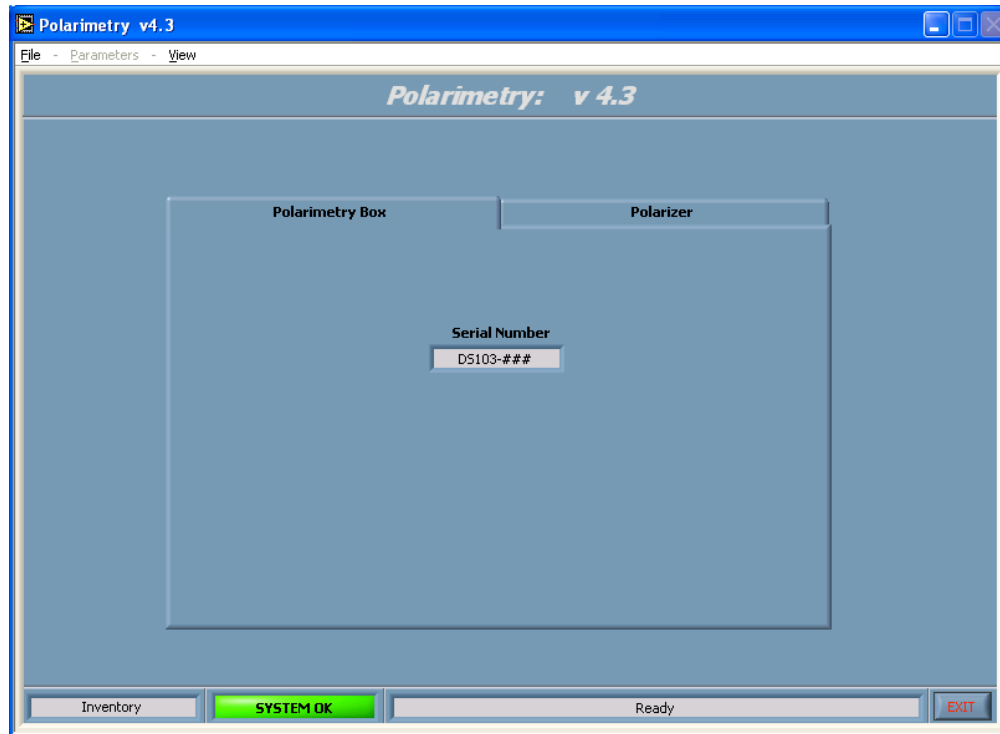


Figure 13: Polarimetry application-Inventory view

The Inventory view contains two tabs. The Polarimetry Box tab displays the serial number of the polarimetry hardware. The second tab, depending on the system version will be either Polarizer, or Polarimetry Station. It displays the serial number of the respective hardware.

Viewing Polarimetry Configuration

The Configuration view displays information about the polarimetry application itself, both the hardware and the software components.

The information on these views is for viewing purposes only and cannot be changed. A Polarean service representative may request this information, however, if the polarizer experiences technical problems.

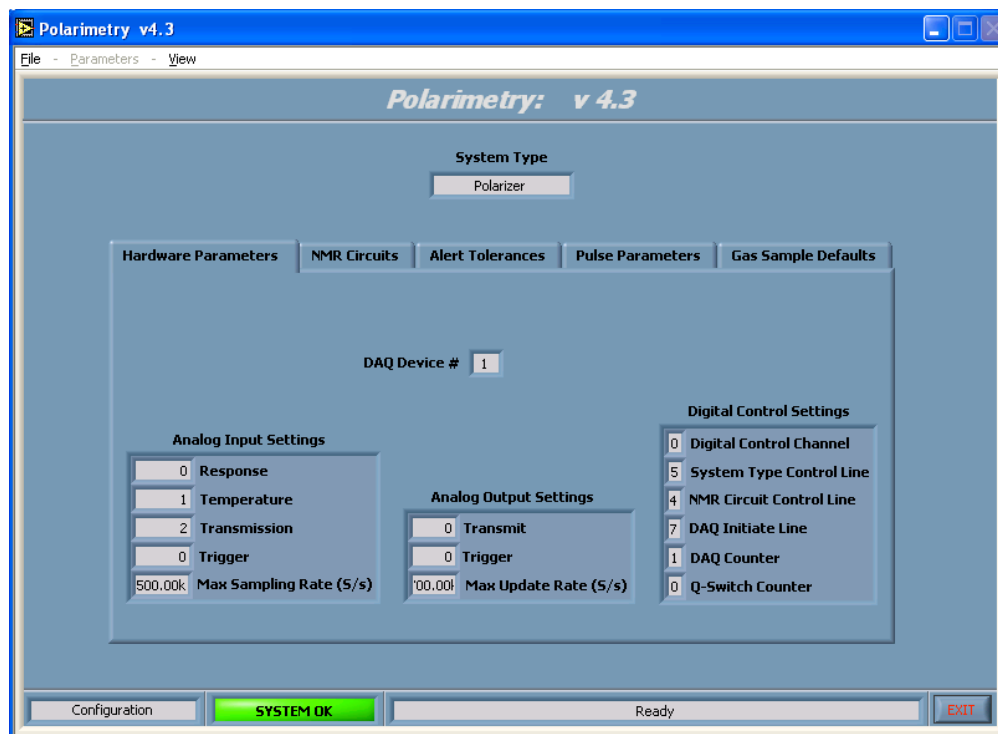


Figure 14: Polarimetry application-Configuration view

The Configuration view contains the following tabs:

Hardware Parameters – Displays the settings used to communicate with the polarimetry box

NMR Circuits – Displays the settings for your machine's NMR (Nuclear Magnetic Resonance) circuits

Alert Tolerances – Displays the diagnostic settings that determine at what level the Resonance Alert and T_2 Alert notifications are activated

Pulse Parameters – Displays the settings that are applied when the pulse parameter from the Pulse Parameters drop-down list box on the Data Acquisition view is selected

Note: This tab displays settings for each pulse parameter option. Selecting a pulse parameter on this tab does *not* change the pulse parameter being used.

Gas Sample Standards – Displays the default settings for Sample Pressure and Sample Blend values

Polarimetry Error Messages

If there is a problem with either the polarimetry application configuration files or any operation involved in determining the polarization percentage, a Failure Mode error message will be displayed, as shown in Figure 15.

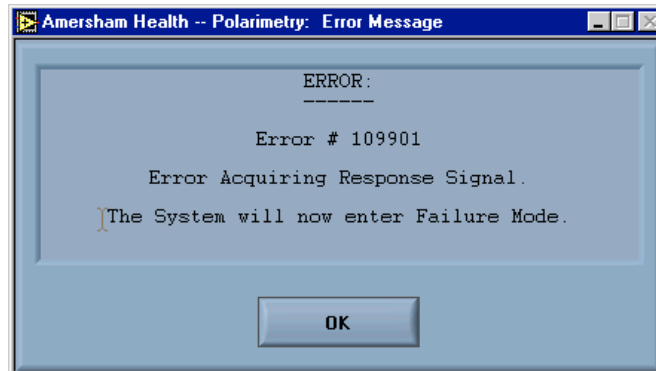


Figure 15: Failure Mode error message

In Failure Mode, the following functionality is unavailable:

- Spin-Up, Spin-Down (current studies are terminated)
- Acquiring data
- Changing Pulse Parameters
- Changing Sample Pressure or Sample Blend values

If a Failure Mode error message is displayed, write down the Error Number and the text contained in the error message, and consult Polarean technical support for instructions.

Revision History

Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.

B

Troubleshooting

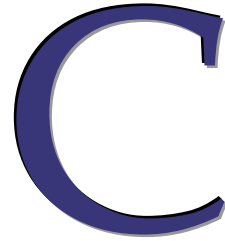
Problem	Solution/Action
Green light(s) on T/I Unit are off, amber warning light(s) on T/I Unit turn on, and/or audible alarm is sounding.	Check interlocks and cables. Check airflow. Flow rate may be too low.
Laser will not turn on.	Verify that the laser interface software is running and the settings are correct. Verify that the front panel key switches are in the On or "Enable" positions. Verify that the Emergency Off button on the laser panel is not depressed (if applicable). Check for error messages on the LCD screen of the laser front panel (if applicable). Verify that the interlock has not been tripped. Verify that the power distribution box is on. Check the interlocks and cables.
Oven temperature is not going up.	Verify the compressed air and heater are on and that the temperature controller is set for a higher temperature than the present temperature. Check to see if the interlock has been tripped. If the air interlock has been tripped, raise compressed air flow rate above 130 SCFH, and then lower to desired flow rate again. Then press the "Reset" button on the temperature-interlock unit.

Problem	Solution/Action
The displays for the digital pressure transducer and/or mass flowmeter on the cartridge are blank.	Verify that the T/I Unit is plugged in and turned on. Verify that the 25 pin cable is connected securely between the T/I Unit and the back of the cartridge
The polarization level of the gas recovered after thawing is lower than expected.	Verify the oven controller maintained a stable temperature (i.e. within $\pm 1^{\circ}\text{C}$ of set point) during accumulation. Check rubidium and cell appearance. Verify the Helmholtz coil power supply is on and at the correct setting. Verify the laser is working and aligned properly. Verify that the laser transmission was in the correct range during collection (<50% for broad and <15% for narrowed lasers). Interval of time may be too long between dispensing gas and signal acquisition (should be under 5 minutes). Collection container may have been left in area of high B field gradients, high magnetic field or near a metal object.
Rubidium in the cell body looks bad (gold color, yellow or brown edges or covered with a white film).	Replace the optical cell. Call Polarean at (919) 206-7900 and ask to speak to technical support. Check for air leaks in the collection container.
Manifold pressure is too high or too low.	Reset the regulator on the Xe gas mixture tank. Verify the pressure in the Xe gas mixture tank is above the regulator set pressure. Verify you have observed the correct valve setup (open valves are open and closed valves are closed).
Pressure in outlet manifold drops during collection.	Increase nitrogen warming jacket flow rate to thaw Xenon ice clog in the cold finger. Check pressure in Xe mixture supply tank and replace tank if necessary.
Laser will not start	Check the Temperature-Interlock Unit. If either

Problem	Solution/Action
	or both of the red interlock lights are on, press Reset.
	Check the room interlock or the room interlock bypass (if installed).
Pressure in vacuum manifold is too high	Verify the vacuum pump is on and <i>Vacuum Isolate</i> valve is open.
	Verify that all valves are in their initial configurations.
	Check Chemthread® connections on cold finger for tightness.
	Gas ballast on vacuum pump should be set to "0".
	Drain any oil collected in the vacuum pump exhaust filter.

Revision History

Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.



Accumulation Data Sheet

The following page contains an optional Accumulation Data Sheet you can use to record the collection information and observe trends in polarizer performance. One sheet can record one or two collections. Print or photocopy this sheet as often as you need to.

Revision History

Date	Change	By whom
5/1/2012	Initial release	Polarean, Inc.

Accumulation Data Sheet

Unique Batch #:		Accumulation Start:		Xenon Blend Cylinder #:		
Date of Production:		Accumulation Finish:		UHP Nitrogen Cylinder #:		
Subject ID:		Operator:		Initial Bottle Pressure:		
Time [HH:MM]	Temperature [°C]	Pressure [psi]	Flow [L/min]	Transmission [%]	NMR	Comments
:						
:						
:						
:						
:						
:						
Post-Thaw		Pressure:	Signal:			
		Signal / P:	Calibration Factor:		[%P / mV @ 1atm]	
			Polarization		[%]	
Comments:						



Polarizer Interlocks

The Polarean Xenon polarizer conforms to the current applicable ANSI standards for a Class 1 laser product. Because there is a Class 4 laser system embedded in the machine, great care should be taken when operating, maintaining and servicing the machine. Protective housing panels designed to be removed by users/operators to perform routine maintenance operations have been fitted with fail-safe keyed interlock switches. Enabling laser emission with the protective housing panels removed could cause severe and permanent damage to eyes and/or exposed skin. *Never* attempt to activate laser emission with any of the panels removed or improperly secured.



Panels designed to be removed only during service operations have been fitted with tamper-resistant screws. *Under no circumstances should anyone but authorized Polarean service personnel attempt to remove the tamper-resistant screws and/or defeat the interlock switches.*

If laser emission is activated with any of the protective housing panels removed or interlocks defeated, the polarizer becomes a Class 4 laser product and must be operated in an environment approved for Class 4 lasers. If any of the protective housing components are removed during maintenance or service, a re-verification test that radiant laser emissions are within Class 1 limits must be performed prior to operating the polarizer in a Class 1 environment.

The interlock switches are wired in series and connected to the External Interlock port of the Temperature/Interlock chassis box. A 2-pin connector is provided in the switch wiring harness to connect the polarizer to an external interlock system. The connector is wired in series with the switches and is supplied with a jumper for use without an external interlock system. Only when both on-board switches are closed (by placing the panels on the polarizer with the keys in the proper slots) and the jumper is attached (or any attached external interlock is closed) will the External Interlock circuit close and allow the laser to enable emission.

Removing and Replacing Interlocked Panels

The interlocked panels are the cart end panel, which is removed when the helium supply cylinder is replaced, and the oven cover panel, which is removed to check or replace the optical cell.

Cart End Panels

To remove the cart side panel:

1. Hold the panel in position while the screws securing it are removed.
2. Pull the panel straight off the end without bending or twisting.

To replace the cart end panel:

1. Hold the panel so that the key is at the bottom and facing the polarizer.
2. Line the key up with the slot in the switch, and push the panel straight on. (A distinct click will be heard as the switch is engaged.)
3. Hold the panel in place until all of the screws securing it have been tightened.

Oven Covers

To remove the oven cover panel:

1. Remove the retaining screws.
2. Lift the cover straight up until the key has disengaged from the switch.

To replace the oven cover:

1. Line the key up with the slot in the switch, and push the cover down until the key is fully inserted.
 2. Replace the retaining screws and tighten.
-

When either of the interlocked panels is removed, a radiant laser emissions check must be performed before returning the unit to operation outside a Class 4 laser environment. See the procedure for Verifying Beam Path Protective Housing Integrity (Radiant Power Emission Check) in Chapter 6.

Interlock Troubleshooting

Problem	Solution/Action
Green light(s) on T/I Unit are off, amber warning light(s) on T/I Unit turn on, and/or audible alarm is sounding.	Press Reset button on T/I Unit Check airflow. Flow rate may be too low. Verify wiring harness for panel switches is plugged into the External Interlock port on the back of the T/I Unit. Verify the 2-pin jumper is plugged into the interlock wiring harness or that any external interlock attached is closed. Verify the interlocked panels are properly affixed to the polarizer with the keys in the slots of the interlock switches.
Laser will not turn on.	Verify that the laser control software is running and the settings are correct. Verify that the "Emergency Off" button is not pushed in on the control panel. Verify that the interlock has not been tripped (interlock indicators on T/I Unit should be in Normal condition). Verify that the power distribution box is on. Verify the laser control panel(s) is/are properly connected to the T/I Unit. Check the interlocks and cables.

Revision History

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5/1/2012	Initial release	Polarean, Inc.



Rubidium MSDS

SECTION 1: PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME: RUBIDIUM (99.9+%) (prescored ampoule)
SYNONYM: Alkali metal
PRODUCT CODES: Rb
MANUFACTURER: ATLANTIC METALS & ALLOYS
ADDRESS: P.O. BOX 589 STRATFORD, CT 06615
EMERGENCY PHONE: 203-378-9025
CHEMTREC PHONE: 800-424-9300
FAX PHONE: 203-378-9570
CHEMICAL NAME: Rubidium
CHEMICAL FAMILY: Metal or element
CHEMICAL FORMULA: Rb

SECTION 2: COMPOSITION/INFORMATION ON INGREDIENTS

CAS NO.: 7440-17-7

SECTION 3: HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: The metal rapidly forms caustic and highly corrosive alkali hydroxide with evolution of heat. Severe thermal and chemical burns will result at every point of human contact

ROUTES OF ENTRY: Skin and eye contact, ingestion

POTENTIAL HEALTH EFFECTS

EYES: Severe thermal burns, corrosion and ulceration of the eyes may occur on direct contact

SKIN: Severe thermal burns, corrosion and ulceration of the skin may occur on direct contact

INGESTION: Will cause burns and perforations of the gastrointestinal tract

INHALATION: Not a likely mode of entry

ACUTE HEALTH HAZARDS: With moisture or human contact, metal rapidly forms caustic and highly corrosive alkali hydroxide with evolution of heat. Severe thermal and chemical burns will result.

CHRONIC HEALTH HAZARDS: No information available on long-term chronic effects.

CARCINOGENICITY

OSHA: No

NTP: No

IARC: No

SECTION 4: FIRST AID MEASURES

EYES: Immediately flush the eyes with copious amounts of water for at least 10-15 minutes. Subject may need assistance in keeping their eye lids open. Seek medical assistance immediately

SKIN: Wash the affected area with water for at least 15 minutes. Remove contaminated clothes if necessary. Seek medical assistance if irritation persists.

INGESTION: Seek medical assistance immediately. Keep the subject calm. If subject conscious, give water. Do not induce vomiting unless directed to do so by medical personnel.

INHALATION: (For dust or fume) remove the subject to fresh air. Closely monitor for signs of respiratory problems, such as difficulty in breathing or coughing. In such cases seek medical assistance immediately.

SECTION 5: FIRE-FIGHTING MEASURES

FLAMMABLE LIMITS: Not Applicable

FLASH POINT: Not Applicable

AUTOIGNITION TEMPERATURE: No data

EXTINGUISHING MEDIA: Class D metal fire agent, dry salt or sand

SPECIAL FIRE FIGHTING PROCEDURES: If this product is involved in a fire, fire fighters should be equipped with a NIOSH approved positive pressure self-contained breathing apparatus.

UNUSUAL FIRE AND EXPLOSION HAZARDS: Pyrophoric solid. May self-ignite. Evokes flammable hydrogen with water.

HAZARDOUS DECOMPOSITION PRODUCTS: If involved in a fire, burning metal may produce irritating fumes of calcium oxide.

SECTION 6: ACCIDENTAL RELEASE MEASURES

ACCIDENTAL RELEASE MEASURES: In case of a spill, the material should be covered with dry soda ash, limestone, or sand. Place in open container. Flammable hydrogen evolution may occur if wet.

SECTION 7: HANDLING AND STORAGE

HANDLING AND STORAGE: Handle and store in a tightly sealed container under an inert atmosphere of argon.

SECTION 8: EXPOSURE CONTROLS/PERSONAL PROTECTION

VENTILATION: Handle in an efficient fume hood

RESPIRATORY PROTECTION: If ventilation is not available a respirator should be worn. The use of respirators requires a Respirator Protection Program to be in compliance with 29 CFR 1910.134

EYE PROTECTION: Always wear approved chemical splash proof goggles

SKIN PROTECTION: Wear protective clothing and gloves. Consult with glove manufacturer to determine the proper type of glove.

OTHER PROTECTIVE CLOTHING OR EQUIPMENT: Prepare for the possibility of fire.

WORK HYGIENIC PRACTICES: Follow usual precautions for handling chemicals. Do not use near any type of food, feed, tobacco, or beverage. Remove all soiled and contaminated clothing prior to leaving work. Wash hands before eating, smoking, or using bathroom facilities, before breaks and at the end of work.

EXPOSURE GUIDELINES: Remove all soiled and contaminated clothing immediately.

SECTION 9: PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE: under argon; silver metallic solid

ODOR: Odorless

PHYSICAL STATE: solid

pH AS SUPPLIED: Not applicable

BOILING POINT:

F: 1270.4°

C: 688°

MELTING POINT: No data
VAPOR PRESSURE (mmHg): No data
SPECIFIC GRAVITY (H₂O = 1): 1.532
SOLUBILITY: Reacts violently with water
MOLECULAR WEIGHT: 85.47

SECTION 10: STABILITY AND REACTIVITY

STABILITY: Moisture sensitive, pyrophoric

CONDITIONS TO AVOID (STABILITY): Contact with water or air

INCOMPATIBILITY (MATERIAL TO AVOID): Water, alcohols, oxidizers, oxygen, carbon dioxide, halogens, halocarbons, acids.

HAZARDOUS DECOMPOSITION OR BY-PRODUCTS: Flammable hydrogen with moisture, caustic oxide with air or moisture

HAZARDOUS POLYMERIZATION: Will not occur

SECTION 11: TOXICOLOGICAL INFORMATION

TOXICOLOGICAL INFORMATION: Intraperitoneal (mouse) LD₅₀: 1200 mg/kg

SECTION 12: ECOLOGICAL INFORMATION

ECOLOGICAL INFORMATION: No information available

SECTION 13: DISPOSAL CONSIDERATIONS

WASTE DISPOSAL METHOD: Dispose of according to local, state and federal regulations

SECTION 14: TRANSPORT INFORMATION

U.S. DEPARTMENT OF TRANSPORTATION
PROPER SHIPPING NAME: Rubidium
HAZARD CLASS: 4.3
ID NUMBER: UN#: 1423
PACKING GROUP: I

LABEL STATEMENT: Dangerous when wet

SECTION 15: REGULATORY INFORMATION

U.S. FEDERAL REGULATIONS

TSCA (TOXIC SUBSTANCE CONTROL ACT): All components of this product are listed in the U.S. Environmental Protection Agency Toxic Substances Control Act Chemical Substance Inventory

313 REPORTABLE INGREDIENTS: Not listed

SECTION 16: OTHER INFORMATION

DISCLAIMER: This information should be used only as a supplement to other information. This information is furnished without warranty.

The use of the product in non-conformance with this Material Safety Data Sheet, or in combination with any other product or process is the responsibility of the user.

Source: Atlantic Metals & Alloys, LLC
100-D Benton St, Stratford, CT 00615
MSDS DATE: **09/01/2011**



The above information is believed to be correct, but does not purport to be all inclusive and shall be used only as a guide. Polarean shall not be held liable for any damage resulting from handling or from contact with the above product.

Revision History

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